51st ANNUAL MEETING OF THE AMERICAN LEATHER CHEMISTS ASSOCIATION

THE JOURNAL

OF THE

AMERICAN LEATHER CHEMISTS ASSOCIATION

VOL. L MARCH 1955 No. 3

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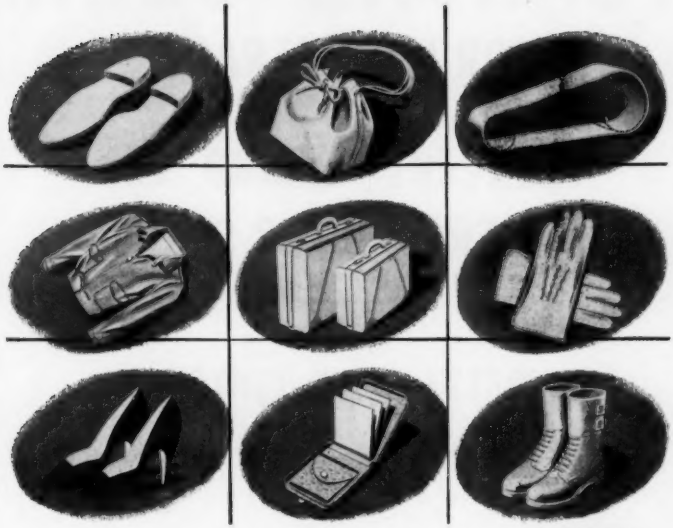

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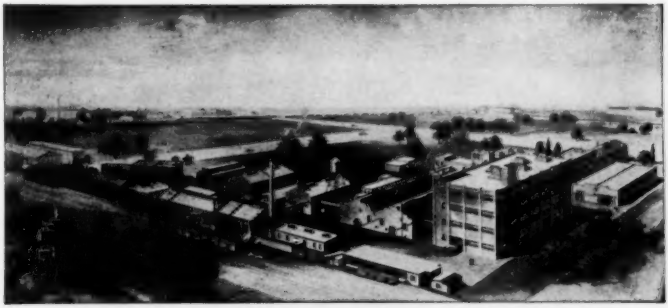

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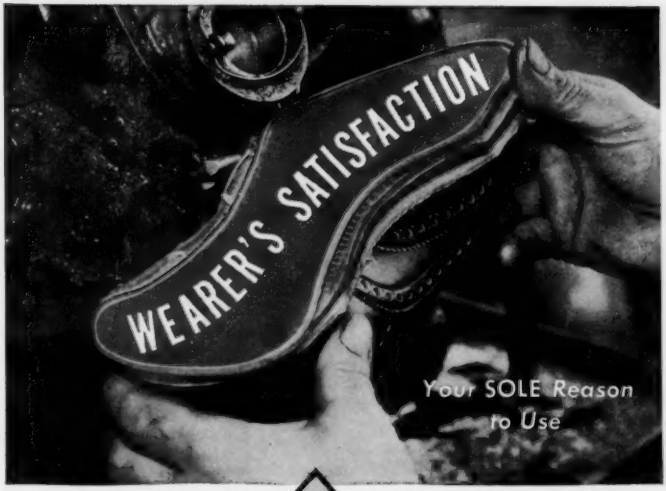

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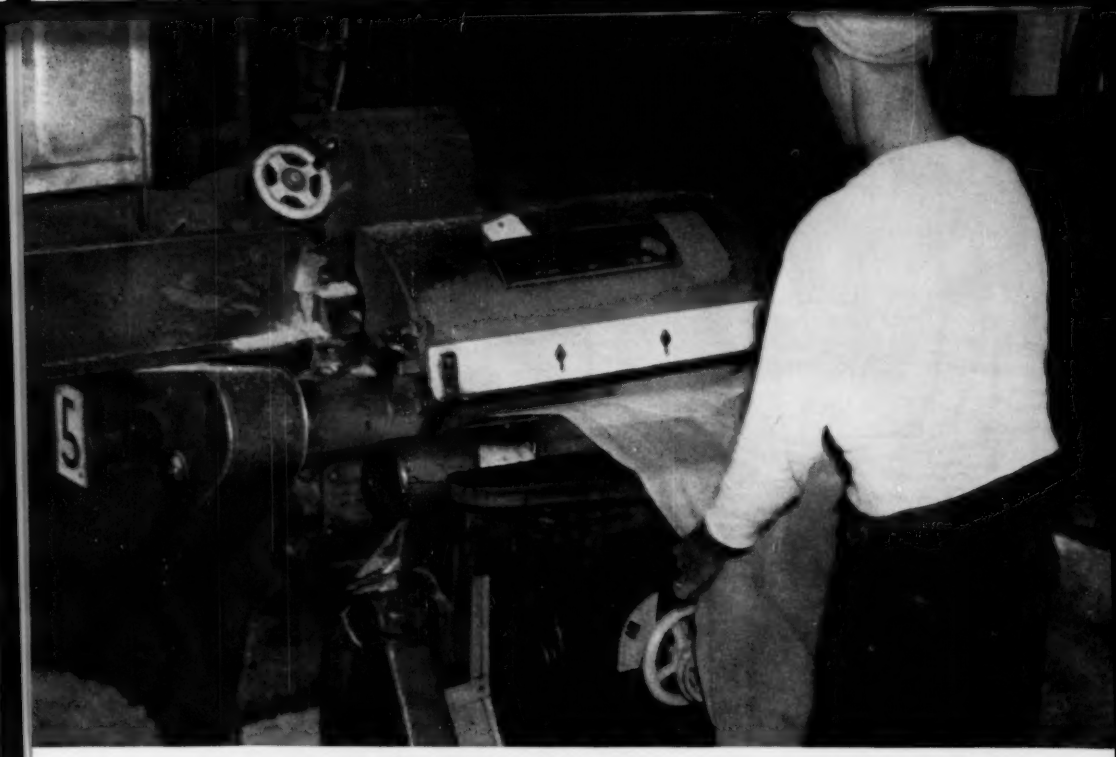

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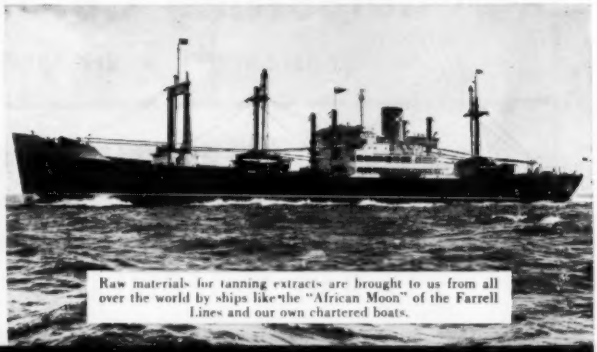

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JOURNAL OF THE
AMERICAN LEATHER CHEMISTS ASSOCIATION

Vol. L MARCH 1955 No. 3

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NOMINATION FOR COUNCIL

The following candidate has been nominated for Council by petition as provided in Article V, Section 2 (c).

ROBERT G. WELDON

NOTICE OF ERROR

The February issue of the 1954 Journal ends with page 132 and the March issue begins with page 173. No material was omitted from the Journal; only paging error exists.

BUREAU OF EMPLOYMENT

LEATHER CHEMIST: With industrial experience offers his services as a tanner or to an industry of leather chemicals. Would work as a tanner's assistant. Can work in research. Academic background. 28 years old. Location secondary. Reply to Box JB103, Dr. Fred O'Flaherty, American Leather Chemists Association, University of Cincinnati, Cincinnati 21, Ohio.

PROVISIONAL PROGRAM FOR STOCKHOLM CONFERENCE OF THE INTERNATIONAL UNION OF LEATHER CHEMISTS SOCIETIES

AUG. 1st. - AUG. 4th, 1955

VENUE OF CONFERENCE

Kungl. Tekniska Hogskolan (Royal Technical Institute), Valhallavagen, Stockholm O.

SUNDAY, JULY 31st.

Afternoon At 5 p. m.—Meeting of Executive Committee of the International Union of Tekniska Muscet. (The Technical Museum).

Evening At 7–10 p. m.—Registration and Reception of Delegates and Visitors by invitation of the Nordic Leather Chemists Society at Tekniska Museet (The Technical Museum).

MONDAY, AUGUST 1st.

Morning 9–10 p. m.—Meeting of Delegates of International Union.
10–12 noon—Official Opening of Conference.
Presidential Address.

Afternoon 1–5 p. m.—Symposium on "The Properties of Leather". Introduction by the Symposium Chairman, Mr. J. R. Blockey. Lectures by Drs. Baumann, Kanagy and Mitton. Discussion. Meeting of Commissions.

Evening—Evening free.

TUESDAY, AUGUST 2ND.

Morning 9 a. m.–12 Noon—Symposium on "Fundamentals of the Chemistry

of Tanning Processes". Introduction by the President of the Union. Lectures by Drs. Pankhurst, Balanyi, White, Kuntzel, Otto and Turley.

Afternoon 1–4 p. m.—Continuation of the Symposium. Discussions on the lectures will be opened by appointed speakers.

Evening at 5 p. m.—Reception at Stadshuset, The Town Hall of Stockholm, by invitation of the Stockholm City Council.

WEDNESDAY, AUGUST 3rd.

Morning 9–12 a. m.—Reading and discussion of scientific papers.
12–1 p. m.—"Our Present Knowledge of Collagen", lecture by Professor W. Grassmann.

Afternoon 2–5 p. m.—Visit to Skansen, The National Folk Park of Stockholm, where lunch will be served followed by a conducted tour of Skansen, including a visit to the old Tannery.

Evening 5–9 p. m.—Excursion around the Archipelago of Stockholm. The Lunch and Excursion are by invitation of the Swedish Leather Industry and Allied Industries.

THURSDAY, AUGUST 4th

Morning 9.30 a. m.–12 Noon—Reading and discussion of scientific papers.

Afternoon 1–4 p. m.—Scientific papers. Open meeting of Conference to receive reports from Commissions. General Meeting. Closing of Conference.

Evening 7 p. m.—Banquet in Gyllene Salen, Stockholm's Stadshus (Town Hall). (Dinner jacket or dark suit).

NOTE: Arrangements have been made for lunch to be provided on Mon., Tues., and Thursday at the nearby restaurant in the Students Hostel "Domus."

A special program will be arranged for the Ladies.

Intending visitors to the Conference are reminded of the desirability of notifying the secretaries of their respective Societies as *soon* as possible, in view of the great demand for hotel accommodation in Stockholm, and in order to facilitate the arrangements. Secretaries of the Societies can then keep the Organisers of the Conference in Stockholm advised.

The official travelling Agents for the Conference are Messrs. Thos. Cook & Son Ltd.

Significance of the Results of Some Physical Tests on Upper Leather*

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ABSTRACTS

The results of various physical tests, including tensile, stitch tearing, tongue tearing, and bursting strength, obtained on chrome and chrome-retanned upper leather are discussed. The tests are compared and correlation coefficients for the variations between the results of tests over the area of a side are given.

From the results, it is concluded that the tensile strength is not influenced by the combination tannage of vegetable tannins and chrome, but that tongue tearing strength and bursting strength are influenced.

It is further concluded that the tongue tearing and bursting strength tests are the best to use for the evaluation of upper leather, since they appear to give an indication of the magnitude of the forces of attraction between fibrous structural units. The tensile strength test gives a measure of the longitudinal strength of these units. The stitch tearing test gives an indication of the load supported by the leather before a tear starts. After a tear develops, the mechanism of the test is similar to the tongue tearing test. Some thought should be given to the conversion of physical strength data to a unit hide substance basis if the results are to be used for making comparisons between different tannages.

1. INTRODUCTION

Leather is composed principally of random interweaving collagen fibers or bundles containing fibers. The condition of these and the manner in which they interweave to form a complex fiber matrix constitutes the fibrous structure of the leather. The physical properties of a leather are the resultant of the physical properties of these fibers so that the fibrous structure is closely related to the strength and other properties of the leather.

The fibrous structure of leather is known to vary over the area of a hide [1]. The fibers are known to be oriented longitudinally to different degrees depending upon the location on the hide and also transversely or perpendicular to the grain surface. The direction of orientation has an important influence on strength [1]. The degree with which the larger fibers are broken and separated into smaller fibrils has an influence on the strength and other physical properties of leather.

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During the past few years much information has been obtained at the National Bureau of Standards on the variation of the strength of leather over the area of a hide [2]. These strength data were obtained by means of the conventional physical strength methods currently applied to leather, namely, the tensile test, stitch tearing test, tongue tearing test, and the bursting test. The strength determinations were made in two directions, parallel and perpendicular to the backbone line. All of the tests indicated that there was considerable variation in strength over the area of a hide. However, in the same or similar locations the strength measurements by the various test methods differed considerably and high figures obtained by one method did not necessarily assure the same to be the case regarding any one of the other test methods. The strength of leather as determined is apparently a function of both the fibrous structure and the mechanism of the test. The purpose of this paper is to point out some of the pertinent relations among the test results obtained and to discuss their meaning with regard to the fibrous structure of leather.

2. Source Of Data

The data in this paper were obtained from tests on 30 split and 30 unsplit chrome-tanned steer sides and 30 chrome-retanned sides consisting of 15 cow and 15 steer sides. Most of these data are presented in four other papers [1, 2, 3, 4], together with the methods used in taking the test specimens and other details.

3. Results And Discussion

The breaking load measured in any strength test on leather is directly related to the number of fibers which become involved in the test prior to rupture. A high strength value for leather, therefore, may result from a large number of fibers, whereas a low value may be due to a rupture that involves only a few fibers. For example, the influence of high grease content and the effect of rate of loading on strength may be mentioned [3]. Lubrication may be considered to permit the fibers to slip over each other more easily to new positions before rupture and, therefore, to distribute the load over more fibers. Likewise, slow application of the load may be considered to permit time for the fiber matrix to rearrange and to distribute the load more uniformly [4].

The method of applying the load on the fibers also influences the measured strength. For example, it has been shown [5] that the stitch tear values obtained are not a true function of thickness as are the tensile values. This is interpreted as due to the fact that the load in the former test is not distributed uniformly over the entire cross section. The fibers on the side of the specimen next to the wire—the grain surface—are stretched more than those on the

flesh side; consequently, the fibers that are stretched most will tear first and, the tear proceeds from the grain towards the flesh. The load recorded in this test is related to the number of fibers that become involved at the moment of rupture. Therefore it is conceivable that in this test a tearing load will be obtained which does not depend entirely on thickness. Similarly, it may be seen that the tongue tear and the bursting tests give entirely different results for the strength of the leather, and these values may require a different interpretation from that of either the tensile strength test or the stitch tear test. Therefore, it may be concluded that the measured strength of leather depends on the mechanism of the test and method of application of the load.

3.1 *Apparent Density*

For a heterogeneous material having a porous matrix, such as leather, it might be expected that there would be some relation of strength to the amount, weight, or apparent density of the fiber matrix in each particular location.

TABLE I

Apparent Densities of Leathers Containing Grease and with Grease Removed
(Values are averages of 4 specimens, each from a separate side)

	Chrome-tanned		Chrome-retanned			
			Cows		Steers	
Location	with grease	degreased	with grease	degreased	with grease	degreased
1	0.8401	0.5976	0.7846	0.5851	0.7828	0.5687
2	0.8141	0.6020	0.7982	0.5684	0.774	0.5680
3	0.7941	0.6260	0.8144	0.6186	0.8016	0.6110
4	0.7938	0.6429	0.8189	0.6174	0.8210	0.6261
5	0.7953	0.6476	0.8356	0.6378	0.8084	0.6315
6	0.7751	0.6322	0.8161	0.6622	0.7962	0.6576
7	0.7904	0.6358	0.8030	0.6526	0.7844	0.6494
11	0.8615	0.6030	0.8140	0.5582	0.7976	0.5722
12	0.8595	0.6297	0.8388	0.6076	0.8036	0.5869
13	0.8201	0.6420	0.8331	0.6312	0.8340	0.6210
14	0.8084	0.6419	0.8472	0.6294	0.8090	0.6468
15	0.8139	0.6438	0.8398	0.6304	0.8208	0.6338
16	0.7947	0.6589	0.8370	0.6368	0.8126	0.6513
17	0.7672	0.6332	0.8224	0.6544	0.8008	0.6396
21	0.8817	0.5804	0.8348	0.5907	0.7961	0.5737
22	0.8832	0.5954	0.8388	0.6127	0.8416	0.6086
23	0.8307	0.6352	0.8407	0.6380	0.8336	0.6226
24	0.8227	0.6449	0.8677	0.6470	0.8621	0.6379
25	0.8160	0.6470	0.8628	0.6396	0.8506	0.6123
26	0.8246	0.6317	0.8577	0.6260	0.8462	0.6228
27	0.8357	0.6095	0.8278	0.6388	0.8300	0.6268
Average	0.8201	0.6276	0.8301	0.6229	0.8147	0.6175

It is assumed that the real density of the fibers is constant. Accordingly, measurements of the apparent density of chrome and chrome-retanned leathers were made from calculations based on the weights and dimensions of the pieces. These determinations were made on degreased specimens and also on specimens containing the original grease placed in the leather by the tanner. The apparent densities of the specimens containing the grease obviously show no relationship to the apparent density of the fiber matrix of the leather; therefore, it was necessary to remove the grease to obtain pertinent values. The correlation coefficient of the apparent density of greased specimens versus grease content for the split chrome-tanned leather was 0.9, which indicated that the apparent density was closely related to the grease content. Results for the apparent densities of chrome-tanned leather and chrome-retanned cow and steer sides are given in table 1. These results are averages of four specimens, one from each location of four different hides.

Although the retanned leathers contain about 20 per cent of vegetable tanning material in addition to the chromic oxide, which is about equivalent to that held by the chrome leather[4], the average apparent density of the former is no greater than that of the latter. It must be concluded, therefore, that a volume change has occurred in the chrome-retanned leather and the amount of hide substance per unit volume has been decreased accordingly. It might then be expected that the strength per unit thickness for the retanned leathers should be lower than that of the chrome-tanned leathers.

To determine whether there is any correlation between apparent density and strength values, coefficients of correlation of the density of the degreased split chrome-tanned leathers and the degreased chrome-retanned cow sides with the physical tests were determined. These coefficients of correlation were calculated for the perpendicular direction and for the sum of the strengths in the perpendicular and parallel directions. The latter parameter was used because the apparent density gives the total weight of the fiber in both directions. The results are given in table 2.

TABLE 2

Correlation of Physical Tests with apparent Density of Degreased Leather

Leather	Direction	Tensile	Stitch tear	Tongue tear	Burst
		r	r	r	r
Split, chrome tanned	Perpendicular	0.42	−0.03	−0.24	0.12
do.	Perpendicular + parallel	0.47	−0.15	−0.23	
Cows, chrome-retanned	Perpendicular	0.27	0.12	0.008	0.13
do.	Perpendicular + parallel	0.36	0.01	−0.022	

These correlation coefficients are too low to be of significance, and therefore it is concluded that there is no relation between apparent density and strength. Tensile strength has the highest correlation coefficient with density, but this also is not sufficiently high to be statistically significant. Since weight or the number of fibers per unit volume is not related to strength, it is assumed that structure is the more important factor determining strength for leather.

3.2 *Tensile Strength.*

Tensile strength was the first strength test applied to leather and at the present time it is more generally applied than any other of the mechanical tests used. In table 3 the tensile strengths of a large number of materials,

TABLE 3

Tensile Strengths of Various Materials

Material	Tensile Strength Lb/in.^{2}
Cast Iron	14,000 to 28,000
Hardened steel spring	85,000 to 427,000
Copper Wire	64,000 to 71,000
Aluminum	28,000 to 43,000
Lead	2,800 to 3,600
Wood	8,500 to 17,000
Silk	50,000 to 85,000
Cotton	28,000 to 114,000
Irish Flax	Greater than 140,000
Viscose	6,000 to 20,000
Well-oriented viscose	57,000 to 114,000
Acetate sheet	5,700 to 13,000
Well-oriented acetate rayon	57,000 to 140,000
Leather	4,000 to 6,000
Kangaroo tendon	47,000 to 58,000

including metals and fibers are given [6]. It may be observed that only the best steels have a higher tensile strength than some of the oriented fibers. Leather, which is a type of woven fabric, has a rather low tensile strength compared to that of a material containing oriented collagen fibers such as kangaroo tendon or certain of the textile fibers.

Breakage in the tensile test may be considered to result from either the rupture of chains or the slipping of adjacent chains. The reason for the high tensile strength of the native fibers is believed to be their predominately primary valence chain structure [6]. A large amount of energy is required to break such chains. In well-oriented artificial fibers it is supposed that the chains that are of limited length are associated over part of their length to

form a true lattice as a result of the interaction forces between hydoxyl and peptide groups. Rupture in such cases may occur as a result of the fracture of the chains or slipping of the associated chains over one another or both. In artificial silk prepared from material that has been carefully treated, in which the orientation is good, the internal cohesion is so considerable that in practice chain fracture only is believed to occur, a condition simliar to that existing in the native fiber[6]. The tensile strengths of well-oriented artificial fibers, therefore, may approach the strengths of native fibers as an upper limit. On slipping, irreversible deformation is believed to lead to thinning of the fiber and ultimately to breakage. A slow application of tension should favor slipping, while a rapid rise in tension should favor rupture of the chains.

For purposes of comparison, kangaroo tendon may be considered as a native, well-oriented collagen fiber. Its strength approaches that of silk and some of the oriented artificial fibers. Therefore it is obvious that the reason leather appears to have such a low tensile strength is because of the arrangement of the fibers in the woven hide matrix. In the determination of the tensile strength of leather using a specimen 0.5 inch wide, it is apparent that the total load is carried largely by the fibers whose directions are nearly parallel to the direction of the load. The tensile strength of leather specimens may be assumed to be proportional to the number of fibers over which the load is distributed.

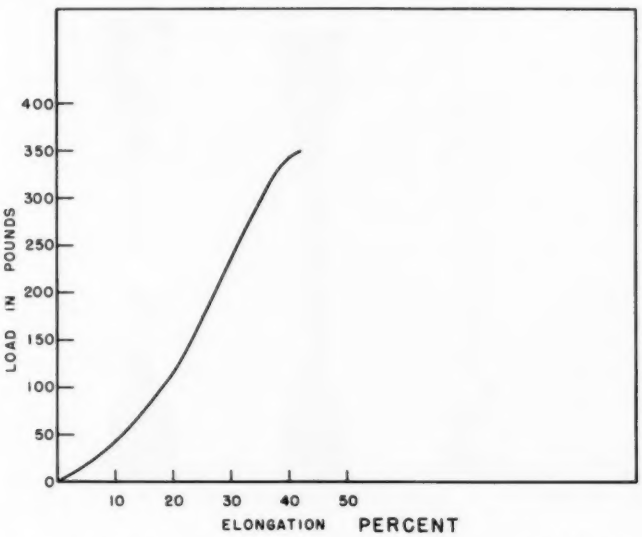


FIGURE 1. Load-elongation curve for tensile strength determination of leather.

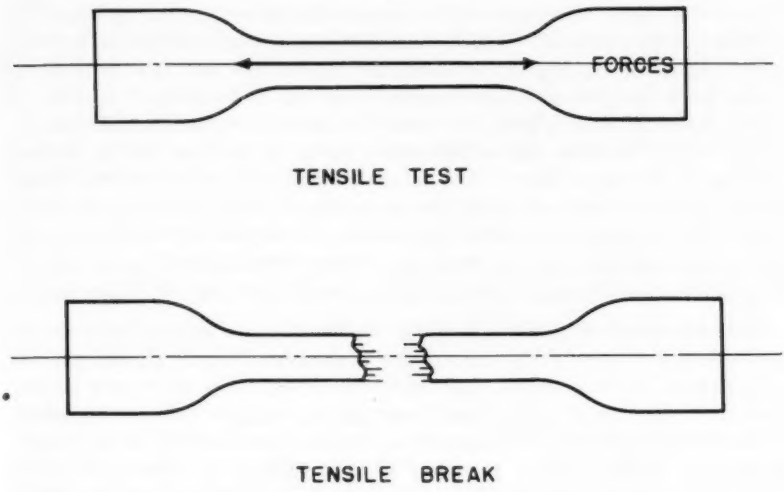


FIGURE 2. Diagram of specimen and forces exerted in tensile strength test for leather.

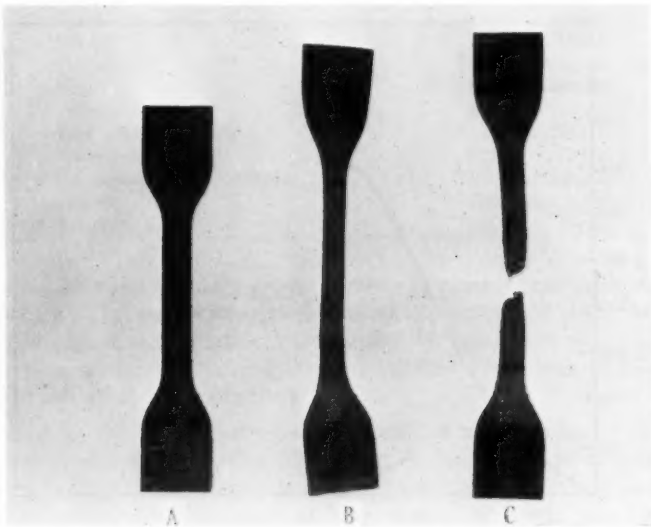


FIGURE 2a. Photograph of specimen used in tensile test. A. Specimen before testing. B. elongated specimen. C. elongated and broken specimen.

The original cross section of the tensile specimen of leather is used to calculate the tensile strength. Because of stretching, which causes narrowing of the cross section, the actual cross section at break is invariably less than that of the original specimen. Stretch is usually determined simultaneously with the tensile strength, but the question of whether or not there is actually any relation between stretch and load at failure has received very little consideration. A typical load-elongation curve for leather is given in figure 1. This sketch shows that considerable elongation occurs before the load on the fibers becomes sufficient to produce rupture. A diagram of the relation between specimen and forces exerted during a tensile test is shown in figure 2. A photograph showing the stretch and final break of a specimen during this test is shown in figure 2a.

By application of a correction based on the stretch, the cross section of the specimen at break may be calculated. The tensile strength may then be based on this figure. Such a procedure has been used by Maron, Madow, and Trinastie [7] and applied to rubber latex films. The equation used was $S = T\frac{(E+100)}{100}$ where S is the actual strength based on the cross section at break, T is the tensile strength calculated on the original cross section, and E is the percentage of elongation. Results calculated on this basis for strengths in the perpendicular direction in all of the positions on one side of split leather are given in table 4. The tensile strength calculated on the original cross section and

TABLE 4

Original and Actual Tensile Strengths and Product of Original Tensile Strength and Elongation of Duplicate Specimens in Various Locations of Chrometanned Steer Side Leather

Location	Original* tensile strength	Elongation	Elongation x original tensile strength	Actual† tensile strength
	10 lb./in.^{2}	%	10 lb./in.^{2}	10 lb./in^2
1	466	65	303	769
	510	67	342	852
2	389	57	222	611
	363	64	232	595
3	354	75	266	620
	420	70	294	714
4	257	53	136	393
	302	70	211	513
5	457	75	343	800
	363	67	243	606
6	469	82	385	854
	720	73	526	1246
7	598	74	443	1041
	546	72	393	939

TABLE 4 (Continued)

Location	Original* tensile strength	Elongation	Elongation x original tensile strength	Actual† tensile strength
	10 lb./in.^{2}	%	10 lb./in.^{2}	10 lb./in^2
11	590	84	496	1086
	598	75	449	1047
12	516	70	361	877
	501	66	331	832
13	558	60	335	893
	558	55	307	865
14	258	78	201	459
	420	66	277	697
15	368	51	188	556
	357	56	200	557
16	391	60	235	626
	390	68	265	655
17	373	68	254	627
	374	71	266	640
21	368	73	269	637
	382	65	248	630
22	298	66	197	495
	351	63	221	572
23	366	57	209	575
	399	76	303	702
24	339	55	186	525
	430	65	280	710
25	520	56	291	811
	559	56	313	872
26	483	70	338	821
	399	73	291	690
27	435	79	344	779
	456	73	333	789
Average	435	67	294	728
Standard deviation	100		78	181
Percentage coefficient of variation	23.0		26.4	24.9

* Tensile strength calculated on basis of original cross section.
† Tensile strength calculated on basis of cross section at break.

the product of the tensile strength and the elongation are also included. The purpose of these experiments was to determine if any of these methods of calculation decreased the variation of the results. Standard deviations and percentage coefficients of variation are given for each of the sets of data. The results show that no improvement is made in the percentage coefficient of variation by basing the strength on the cross section at break or on the product of the tensile strength and the elongation. The results for strength based on the cross section at break are much higher, as is to be expected, but the variability of the results has not been improved.

The tensile strength of a side of leather shows considerable variation depending upon the location on the side or area in which the test is made and also depending upon the direction in which the specimen for the test is cut [1, 2, 4]. A diagram showing the plan for cutting the sides into blocks for these tests is given in figure 3 and described in detail in a previous publication [2]. The

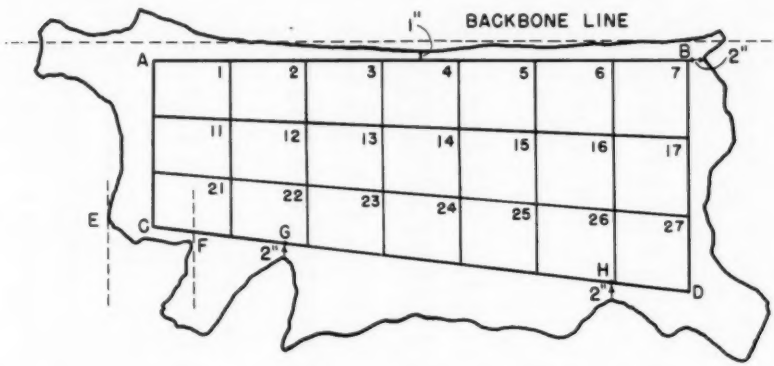


FIGURE 3. Plan used in cutting sides into blocks for the tests.

average tensile strength for a side in these tests is nearly 20 per cent greater if determined parallel to the backbone rather than if determined perpendicular to the backbone. This may be interpreted to indicate that a greater percentage of the fibers are oriented in the parallel direction. The stretch, on the other hand, shows the opposite trend. Stretch is approximately 20 per cent less if measured parallel to the backbone than if measured perpendicular to the backbone. If the product of the stretch and the strength in the two directions is determined, a value nearly constant for both directions is obtained. Such results for five different sets of data are given in table 5. The product of the load and the percentage of stretch gives a measure of the energy of rupture. The results show that on the average the energy of rupture is equal in both directions for the same hide and the same tannage.

Several explanations for this observation may be given. The first is similar to one suggested by Mitton [8] in which it is assumed that the fibers behave as the individual strands in a wire screen or mesh. It is noted that in the performance of the tensile test, the cross section becomes narrower when the load is applied and therefore the fibers tend to become oriented in the direction of the applied stress. This permits the mesh to elongate in a single direction. It may be assumed that the fewer the number of fibers initially oriented in a definite direction, the more the mesh may distort or stretch when pulled in that direction. The magnitude of stretch therefore, would be inversely

TABLE 5

Product of Tensile Strength and Elongation

a. Split chrome-tanned sides*

Side	Tensile Strength // lb./in.²	Elongation // %	Strength x elongation // lb./in.²	Tensile strength ⊥ lb./in.²	Elongation ⊥ %	Strength x elongation ⊥ lb./in.²
A	528x10	60	317x10	449x10	68	305x10
B	464	55	255	391	61	238
C	573	53	304	432	65	281
D	525	53	278	459	62	285
E	475	49	233	389	59	230
F	544	52	283	435	63	274
G	581	53	308	430	64	275
H	520	56	291	416	65	270
J	513	51	262	420	66	277
K	490	53	260	381	70	267
L	516	52	268	417	63	263
M	452	51	230	407	59	240
N	576	53	305	447	65	291
O	526	51	268	391	74	289
P	493	56	276	480	60	288
Q	475	51	242	398	61	243
R	506	51	258	472	60	283
S	554	56	310	449	66	296
T	478	53	253	435	67	291
U	466	52	242	386	63	243
V	438	56	245	388	57	221
W	461	53	244	414	64	265
X	482	52	251	376	68	256
Y	553	54	299	463	67	310
Z	420	58	244	344	68	234
XA	424	53	225	353	62	219
XB	522	55	287	400	71	284
XC	518	54	280	438	62	272
XD	496	55	273	437	67	293
XE	477	55	262	373	67	250
Average			268			268

b. Unsplit chrome-tanned sides*

T	474x10	53	250x10	415x10	68	282x10
W	508	56	284	431	63	272
Y	564	52	293	421	64	269
X	502	53	266	430	64	275
Z	498	50	249	412	60	247
XA	587	49	288	454	65	295

*Results given for parallel direction are averages of 21 to 42 specimens; those in horizontal direction are averages of 42 specimens.

Table 5 (Continued)

Side	Tensile Strength lb./in.² //	Elongation % //	Strength x elongation lb./in.² //	Tensile strength lb./in.² ⊥	Elongation % ⊥	Strength x elongation lb./in.² ⊥
XB	588	49	288	432	67	289
XD	513	50	256	403	62	250
XE	487	47	229	410	61	250
V	476	52	248	380	64	243
U	469	53	249	420	60	252
S	460	46	212	400	55	220
R	504	50	252	398	62	247
XC	520	51	265	384	69	265
B	519	49	254	455	59	268
C	483	55	266	420	69	290
D	548	52	285	471	66	311
E	556	55	306	471	64	301
F	494	53	262	434	65	282
G	448	52	233	387	66	255
A	462	49	226	410	53	217
O	489	56	274	410	64	262
N	492	54	266	380	66	251
H	562	53	298	452	64	289
J	473	55	260	398	65	259
P	467	53	248	388	64	248
L	520	53	276	407	69	281
K	496	56	278	410	64	262
Q	524	50	262	444	65	289
M	500	57	285	403	66	266
Average			264			266
			c. Chrome-retanned cow sides*			
A	400x10	51	204x10	327x10	57	186x10
B	475	52	247	401	56	225
C	427	50	214	361	58	209
D	356	48	171	315	52	164
E	461	52	240	367	60	220
F	378	51	193	326	55	179
G	362	52	188	297	65	193
H	410	50	205	356	57	203
J	364	54	197	293	56	164
K	387	55	213	331	62	205
L	449	49	220	388	56	217
M	377	51	192	335	59	198
N	387	46	178	335	55	184
O	408	50	204	353	54	191
P	395	55	217	335	60	201
Average			206			196

*Results given for parallel direction are averages of 21 to 42 specimens; those in horizontal direction are averages of 42 specimens.

Table 5 (Continued)

d. Chrome-retanned steer sides*

Side	Tensile Strength	Elongation	Strength x elongation	Tensile strength	Elongation	Strength x elongation
	lb./in.²	%	lb./in.²	lb./in.²	%	lb./in.²
	//	//	//	⊥	⊥	⊥
Q	344x10	47	162x10	313x10	50	157x10
R	368	46	169	292	60	175
S	367	44	161	294	53	156
T	333	44	147	320	50	160
U	325	45	146	278	53	147
V	346	52	180	318	60	191
W	416	47	196	337	54	182
X	368	50	184	304	57	173
Y	409	49	200	369	55	203
Z	330	45	149	278	53	147
XA	286	42	120	257	53	136
XB	317	45	143	285	50	143
XC	383	48	184	309	56	173
XD	348	47	164	335	55	184
XE	297	44	131	259	52	135
Average			162			164

e. Chrome-tanned sides used in study on splitting†

A	575x10	51	293x10	443x10	69	306x10
B	590	50	295	442	63	278
C	642	48	308	459	68	312
D	541	57	308	406	74	300
E	612	52	318	495	66	327
F	545	48	262	444	64	284
G	578	54	312	441	68	300
H	567	55	312	444	68	302
J	673	53	357	500	65	325
K	595	49	292	429	71	305
Average			306			304

*Results given for parallel direction are averages of 21 to 42 specimens; those in horizontal direction are averages of 42 specimens.
†Results are averages of 12 to 19 specimens.

proportional to the number oriented in any one direction. On the other hand, strength would be directly proportional to the number of fibers oriented in a given definite direction. Therefore the product of the two should be relatively constant, and for any single hide or group of hides from the same lot this constant should be the same in either direction of test. From these assumptions tensile strength in relation to direction of test depends upon the number of fibers oriented in that direction, and the tensile strength results might be

used to determine directions of orientation. From the results of the tests made in the upper leather study, it is concluded that in the belly and shoulder areas the majority of the fibers are oriented parallel to the backbone, whereas in the butt areas the orientation seems to be about equal in both directions.

Another explanation preferred by some workers is the one based on crystalline and amorphous regions in the fiber. The crystalline regions are those in which it is is assumed that the polypeptide chains are in a high order of alignment and, therefore, would have high strength and very little stretch. In the amorphous regions there is a low order of alignment. Here the chains tend to unfold and the stretch is high while the strength is low. Therefore, the resultant strength and stretch of leather might be assumed to be proportional respectively to the relative number of crystalline and amorphous regions in the fiber. High strength and low stretch is indicative of a large proportion of crystalline regions, whereas low strength and high stretch is indicative of a large proportion of amorphous regions.

A third explanation has a more direct bearing on the energy relations derived from the load-elongation results. It is assumed that the mechanism of rupture consists in part of the rupture of primary valence bonds in the chains and in part of slipping of adjacent chains. Since the greatest amount of energy is probably involved in the breaking of bonds, it is assumed that the magnitude of the tensile strength would be largely dependent on the amount of bond breakage. On the other hand, it is expected that slipping would result in the greatest amount of stretch and the lowest strength. Since the two mechanisms of break have inverse effects upon strength and stretch, it appears probable that the product of the two to the first approximation should equal a constant.

Since the results show that the product of the tensile load at rupture and the stretch is reasonably constant for the two directions when tests are made on the same side, it appears that there is a definite relationship between tensile strength and stretch of leather.

3.3 *Stitch Tear*

This test was introduced recently for the evaluation of leather with regard to strength of seams. It was devised to simulate the behavior of the stitches in shoes under service conditions. It has become quite popular because only a small amount of leather is required to make the test and the test procedure is simple. A diagram of the test as applied to upper leather is shown in figure 4, and is described in Federal Specification KK-L-311a Method 2151 and also in ALCA Booklet, Method F, Fig. E 13. The sketch shows the specimen as used in the test, the direction of the forces exerted in the test proceedure and the type of failure that generally occurs. Two holes, each 5/64 inch in diameter, are punched in one end of a rectangular specimen of leather two

FIGURE 4. Diagram of specimen and forces exerted in stitch tearing test for leather.

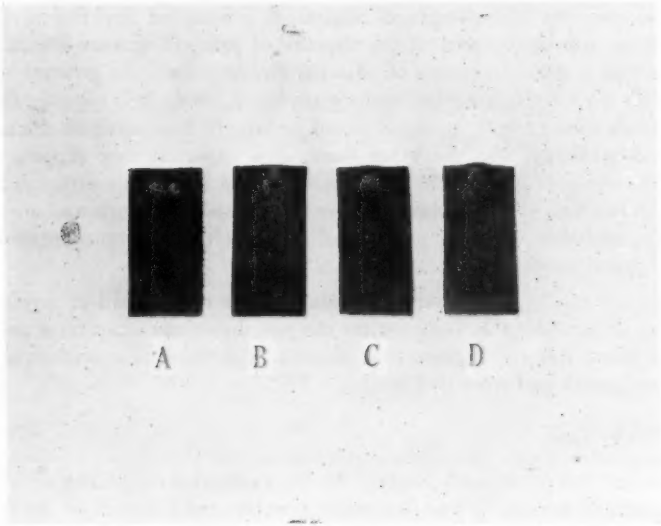


FIGURE 4a. Photograph of specimen in stitch tear test. A, specimen before testing. B, Grain surface of specimen after testing. C, D, Flesh surfaces of specimen after testing.

inches by one inch. The center of each hole is ¼ inch from the end of the specimen and the holes are ¼ inch apart. A soft metal wire, 0.04 inch in diameter, is made into a U-shape and passed through the two holes so that both ends project from the flesh side of the specimen. The ends of the wire

are clamped in one grip of the testing machine and the free end of the specimen is clamped in the other grip. The force is applied so that the power-actuated grip travels at a uniform speed of 10 ± 2 inches per minute. At the moment of the initial tear of the specimen, the force required to start the tear is noted. This value is recorded as the stitch tearing strength of the specimen.

As shown in figure 4 the force exerted is essentially perpendicular to the specimen and the fibers, whereas in the tensile test the force is parallel to and along the specimen and the fibers and in the same direction as the applied load. The photograph (figure 4a) shows that the section of leather between the holes is torn. Although the load is applied perpendicular to the fibers it was expected that there might be some correlation with tensile strength. As may be noted from the mechanism of the test, macroscopic stretch is not expected to play as important a role in the stitch tear test as in the tensile test. From previously published results [2, 4] it may be noted that there is a directional difference in strength for the stitch tear; as with tensile strength the stitch tear strength is greater parallel to the backbone than perpendicular to the backbone. The difference in the strength in the two directions is, however, only about 10 per cent, whereas for tensile strength the directional difference is about 20 per cent.

In some respects the stitch tear test resembles a simple tear test with the tearing force concentrated at two points instead of one. It is similar to a tear also because the load is not applied uniformly over the cross section but the fibers on the grain side next to the wire presumably are stretched more than those on the flesh side and a tear develops at that point. The failure does not occur instantaneously over the entire cross section, but starts on the side where the fibers are pulled to the limit of extension. A reinforcement of fibers, because of stretching, occurs below the hole in the direction of the pull. Failure as a rule does not occur, therefore, by the wire pulling through the leather but by means of a tear which runs in a direction perpendicular to the applied force with the result that a three-cornered piece is pulled out of the specimen. The magnitude of the stitch tear strength, therefore, should depend upon the number of fibers on which the load is distributed before a tear starts. This mechanism has been discussed previously in a paper on the effect of splitting on strength [5].

Insofar as the mechanism of the test requires that the fibers over a ¼ inch wide section must be broken, the load when properly calculated should approach that of the tensile strength; however, since a tear finally develops it appears that it might be related to the tongue tear test. Since the spacing of the holes in the stitch tear test is ¼ inch apart, and the results for the tensile strength are calculated for a 1-inch cross section, the results for stitch tear per inch of thickness when multiplied by 4 should approximate the results obtained for the tensile strength. Likewise, since the stitch tear test tears the

leather, the results for stitch tear might approximate the tongue tearing strength. In order to determine the relation between the different tests, plots were made of the values of any one test versus the corresponding values of any other test (figures 5, 6, 8, 10, 11, 12). There appeared to be no justification for fitting curved lines; therefore, equations representing straight lines for the variation of the results of one test with another were calculated by the least squares method.

Equations representing the variation of stitch tearing strength with tensile strength were calculated from the results of the averages for individual blocks for retanned cow and steer sides in two directions of test and for split and unsplit chrome-tanned leather in two directions of test. The four equations are as follows:

Cows and steers $\perp$ tensile $= 4.33$ stitch tear $- 820$
Cows and steers// tensile $= 2.94$ stitch tear $+ 760$
Split and unsplit $\perp$ tensile $= 2.15$ stitch tear $+ 1510$
Split and unsplit // tensile $= 0.20$ stitch tear $+ 4750$

The variation of the stitch tear with tensile strength is shown in the graphs given in figure 5, and the above equations represent the lines drawn on the

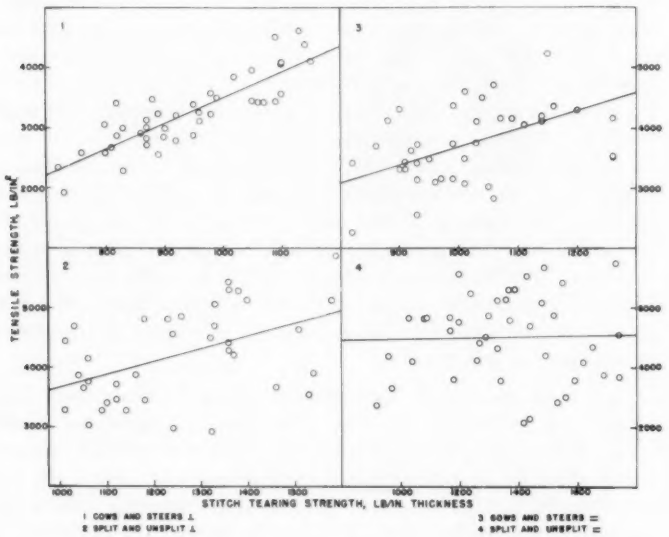

TENSILE STRENGTH, LB/IN²
STITCH TEARING STRENGTH, LB/IN. THICKNESS
1 COWS AND STEERS ⊥
2 SPLIT AND UNSPLIT ⊥
3 COWS AND STEERS =
4 SPLIT AND UNSPLIT =

FIGURE 5. Variation of the sttich tearing strength with the tensile strength for four different groups of leather specimens.

graphs. It may be noted that only one set of results, that for the cows and steers in the perpendicular direction, approaches the theoretically assumed relation, namely, that the tensile strength should be four times greater than the stitch tearing strength. The correlation coefficient for tensile and stitch tearing strength for cows and steers in the perpendicular direction for this set of results is also greater than for any other set of results for tensile and stitch tearing strength as shown in table 6.

TABLE 6

	Side averages*							
	Steers		Cows		Split		Unsplit	
	⊥	//	⊥	//	⊥	//	⊥	//
Tensile vs stitch tear	0.71	0.54	0.68	0.14	0.46	0.28	0.58	0.46
Tensile vs tongue tear	0.64	0.23	0.49	0.64	0.63	0.24	0.65	0.73
Tensile vs burst	0.70	0.55	0.91	0.88	0.73	0.49	0.78	0.33
Stitch tear vs tongue tear	0.84	0.55	0.43	0.39	0.34	0.66	0.73	0.45
Stitch tear vs burst	0.83	0.65	0.77	0.41	0.33	0.46	0.51	0.49
Tongue tear vs burst	0.74	0.70	0.54	0.81	0.66	0.67	0.68	0.70

*—Based on 10 to 42 specimens

	Block averages†							
	Steers		Cows		Split		Unsplit	
	⊥	//	⊥	//	⊥	//	⊥	//
Tensile vs stitch tear	0.87	0.52	0.93	0.88	0.78	0.22	0.32	0.16
Tensile vs tongue tear	0.71	0.15	0.82	0.54	0.62	0.05	0.26	0.39
Tensile vs burst	0.76	0.46	0.78	0.73	0.64	0.41	0.40	0.19
Stitch tear vs tongue tear	0.84	0.71	0.87	0.77	0.87	0.71	0.89	0.79
Stitch tear vs burst	0.78	0.69	0.82	0.68	0.73	0.71	0.69	0.66
Tongue tear vs burst	0.80	0.88	0.75	0.88	0.70	0.73	0.63	0.72

† Based on 7 to 60 specimens.

A comparison of the results in this table with the equations given above indicates as may be expected that the slopes of the lines are related to the correlation coefficients, and as the former decrease, the latter also decrease.

It is concluded from the results of these calculations that no consistent relation exists between stitch tear and tensile strength. The relation varies with both direction of test and the type of tannage.

The variation of stitch tearing strength with tongue tearing strength in the four sets of data was calculated and the equations obtained are as follows:

Cows and steers $\perp$ stitch tear $= 1.05$ tongue tear $+ 500$
Cows and steers // stitch tear $= 0.87$ tongue tear $+ 680$
Split and unsplit $\perp$ stitch tear $= 1.07$ tongue tear $+ 510$
Split and unsplit // stitch tear $= 0.88$ tongue tear $+ 790$

Calculations of precision of the slopes of these equations indicated that the slopes were not significantly different from 1.0. The slope for all sets of data was therefore taken as 1.0 and the intercepts were recalculated. The single equation derived in this way to fit all the different sets of data is as follows:

Stitch tear $=$ tongue tear $+ 606$

The variation of the stitch tearing strength with the tongue tearing strength is shown in figure 6. The line drawn through the points is represented by the above equation.

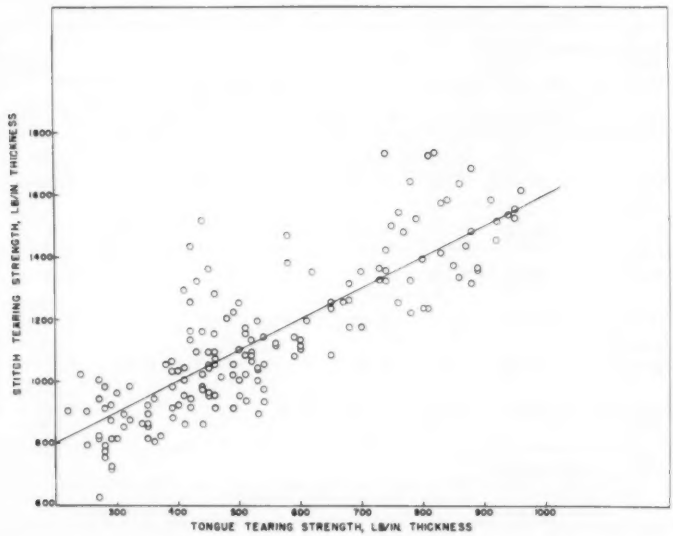


FIGURE 6. Variation of the stitch tearing strength with the tongue tearing strength for leather.

From the results of these calculations it appears that the stitch tearing and the tongue tearing tests have closely related mechanisms. The high value for the intercept 606 indicates that the principal difference is due to the method of application of the test. The wire used in the stitch tearing test makes it possible to support a fairly high load before a tear develops. Part of this load is also involved in the distortion of the leather specimen. The stitch tearing test therefore may be classified as a test which depends to an appreciable extent on the load which may be supported by the leather before a tear is started, and for this reason it is a valuable test in the evaluation of seams.

3.4 *Tongue Tear*

The tongue tear test, as applied to leather in the United States and described in Federal Specification KK-L-311a, Method 2111 or in ALCA Booklet pg. E12 is a simple tear test. A piece of leather about 1 inch wide and 6 inches long is split at right angles to the grain and flesh sides for about two-thirds of its length. The end of one of the pieces formed by the split is put in one jaw of the testing machine and the other end is placed in the other jaw. (See figure 7). The load required to tear the leather is determined.

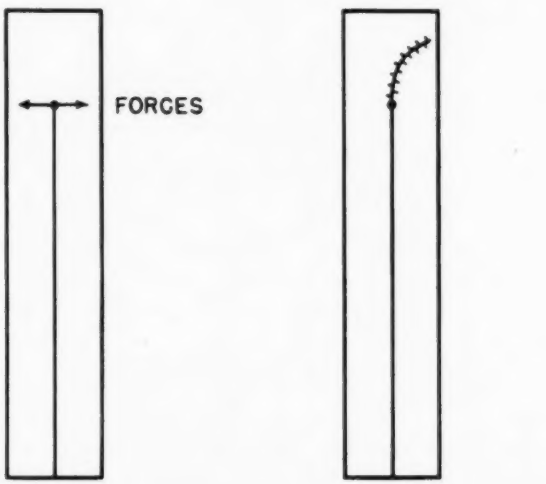


FIGURE 7. Diagram of the specimen and forces exerted in the tongue tearing strength test for leather.

The results obtained from the tests on the leathers used in the sampling study [3] indicate that belly and shoulder areas give, in general, higher tongue tear values than the areas along the backbone and butt. Retanned leather gives lower values for tongue tear than chrome-tanned leather. Tongue tear differs from both the tensile strength test and the stitch tear test in that the strength in the direction perpendicular to the backbone is higher than that in the parallel direction. The reason for this is that the mechanism of the tongue tear test is such that the fibers actually broken are those running perpendicular to the direction of the cutting of the specimens.

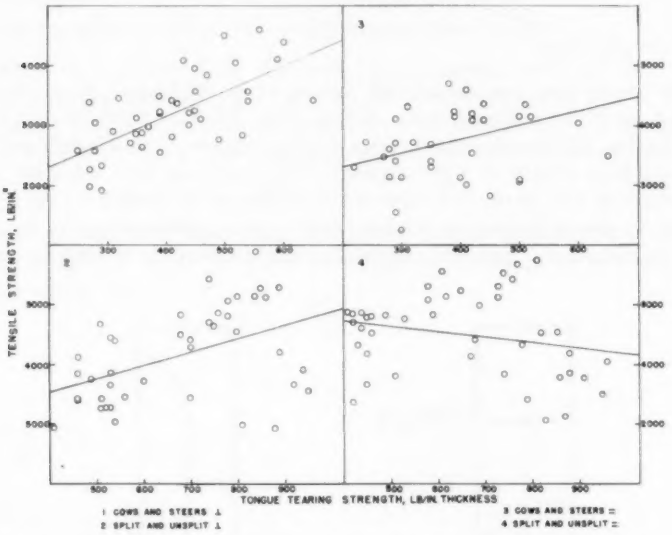

TENSILE STRENGTH, LB/IN²
TONGUE TEARING STRENGTH, LB/IN THICKNESS
1 COWS AND STEERS ⊥
2 SPLIT AND UNSPLIT ⊥
3 COWS AND STEERS //
4 SPLIT AND UNSPLIT //

FIGURE 8. Variation of the tongue tearing strength with the tensile strength test for four different groups of leather specimens.

The variation of tongue tearing strength with tensile strength is shown in figure 8, and the equations for the lines drawn on the graphs are as follows:

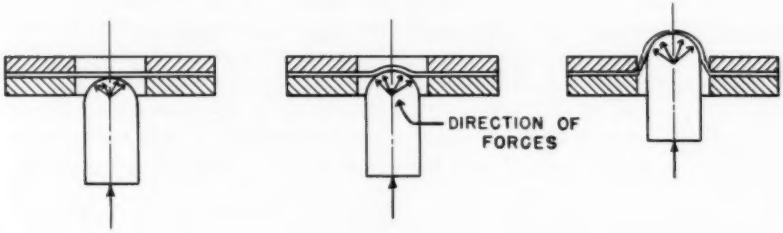
Cows and steers $\perp$ tensile $= 4.25$ tongue tear $+ 1460$
Cows and steers // tensile $= 2.29$ tongue tear $+ 2870$
Split and unsplit $\perp$ tensile $= 2.19$ tongue tear $+ 2680$
Split and unsplit // tensile $= -1.68$ tongue tear $+ 6090$

These equations show that there is no consistent relation between tongue tear and tensile strength. A negative slope is obtained for the split and unsplit leathers for the parallel direction of test. This is in line with the

statement made in the discussion of the mechanism of the test, namely, that the fibers actually broken were those perpendicular to the direction of cutting the specimen*. The relation of tongue tearing strength with tensile strength is similar to the relation of stitch tearing strength and tensile strength. The similarity of the tongue tearing and stitch tearing tests has already been discussed. Correlation coefficients as shown in table 6 for block averages for the two tests are all high.

3.5 *Burst Test*

One of the principal advantages in using the burst test is that it gives an average value of the strength in both directions. A thorough study of the burst test is given in another paper [9], and a diagram of the test and the forces involved are given in figure 9.



BURST TEST

FIGURE 9. Diagram of the specimen shape and forces exerted during the bursting strength test.

In this test a specimen of leather 4 by 4 inches is clamped between two corrugated steel plates having an aperture 1 3/8 inches in diameter. This adapter is placed in one jaw of a testing machine having a loading capacity of 2,000 lb. In the other jaw a plunger, hemispherical at the tip, 2 inches long and 1 inch in diameter is fastened. The force is applied to the plunger so that it moves at the rate of 1 inch per minute. The force required to crack the grain of the leather and that required to burst the leather are recorded.

The burst test is closely related to performance in shoe-making. Pulling the upper over the last applies forces similar to those applied in the test. Since the first failure noted is generally cracking of the grain, it may also be used for measuring the tenderness of the grain. The bursting strength is

*It appears from these considerations that a better correlation might be obtained if the relation between the two tests was determined in directions differing by 90°, namely, tensile strength in the perpendicular direction correlated with tongue tearing strength in the parallel direction. This was done with the result that a slight improvement was obtained with chrome-retanned leather from cow and steer sides and no improvement for the chrome-tanned leather.

expressed in pounds per inch of thickness. The results obtained in the testing of the leather used in the sampling study show that chrome-tanned leather has a higher bursting strength than chrome-retanned leather, and that the belly locations have higher strength than the other locations over the area of the hide.

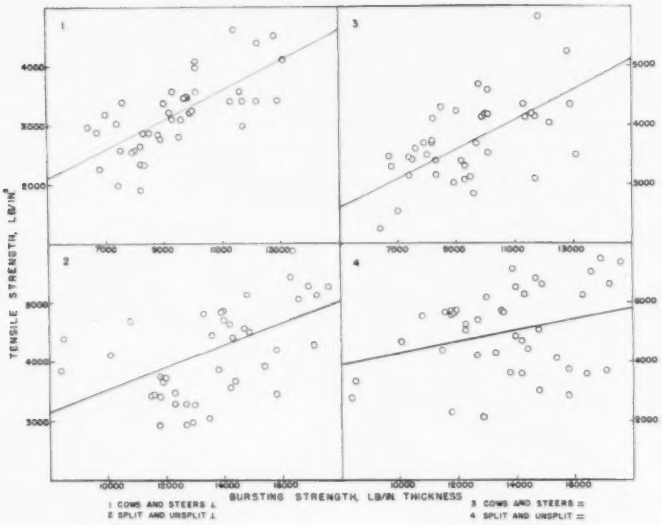

TENSILE STRENGTH, LB/IN²
BURSTING STRENGTH, LB/IN THICKNESS
1 COWS AND STEERS ⊥
2 SPLIT AND UNSPLIT ⊥
3 COWS AND STEERS //
4 SPLIT AND UNSPLIT //

FIGURE 10. Variation of the bursting strength with the tensile strength for four different groups of leather specimens.

The variation of results of tensile strength with those of bursting strength are shown in figure 10, and the equations for the lines shown on the graphs are as follows:

Cows and steers $\perp$ tensile $= 0.25$ burst $+ 860$
Cows and steers // tensile $= 0.25$ burst $+ 1350$
Split and unsplit $\perp$ tensile $= 0.19$ burst $+1610$
Split and unsplit // tensile $= 0.19$ burst $+ 2380$

The slope of the straight lines expressing the relation between burst and tensile strength differs only with tannage and not at all with the direction of pull as figure 10 shows. The same is true of the relations between burst and

stitch tear and between burst and tongue tear (figures 11 and 12). The equations for the lines drawn on the graphs are as follows:

Cows and steers $\perp$ burst $= 11.5$ stitch tear $- 1250$
Cows and steers $//$ burst $= 8.5$ stitch tear $+ 830$
Split and unsplit $\perp$ burst $= 7.87$ stitch tear $+ 3940$
Split and unsplit $//$ burst $= 6.99$ stitch tear $+ 4180$
Cows and steers $\perp$ burst $= 13.9$ tongue tear $+ 3730$
Cows and steers $//$ burst $= 15.4$ tongue tear $+ 3510$
Split and unsplit $\perp$ burst $= 8.98$ tongue tear $+ 7580$
Split and unsplit $//$ burst $= 8.52$ tongue tear $+ 8190$

The abrupt change in the constants obtained in these equations when they represent retan instead of straight chrome would indicate that there is a different relation between stitch and tongue tear on the one hand and burst on the other, dependent on the method of tanning. The same phenomenon has already been observed between tensile strength and burst.

The results of the burst tests consistently show a high correlation with the other three strength tests, but the correlation between burst and tensile strength is always the lowest of these three, and here the block averages are poorer than the side averages as table 6 reveals.

4. Effect of Tannage on Results of Strength Measurements

In figures 13 and 20 the tensile strengths of the chrome-tanned split and unsplit leathers and the retanned cow and steer sides in the two directions of test are compared with the results for burst, stitch tear, and tongue tear. These results are averages for the various blocks on the side. In studying these figures it must be remembered that burst is a nondirectional test. The values for burst appear in different orders on the graph because the block numbers are different. (See figure 3.) The blocks are arranged according to increase in tensile strength. The results for the other tests in the different blocks are then plotted and compared with the tensile strength. These plots give a graphical picture of the correlation of the various tests with each other.

The values for tensile strength show the greatest variation between positions, that is, the line connecting the value for the block showing lowest tensile strength and that showing the highest tensile strength has the greatest slope. The values for the burst test show the next greatest slope. The curve conecting the values for the burst strength rises slightly as the tensile strength increases in all of the figures. Stitch tear and tongue tear in the parallel direction do not increase as the tensile strength increases, and therefore in the parallel direction of test there is poor correlation between these tests. This is confirmed by the calculated correlation coefficients given in table 6.

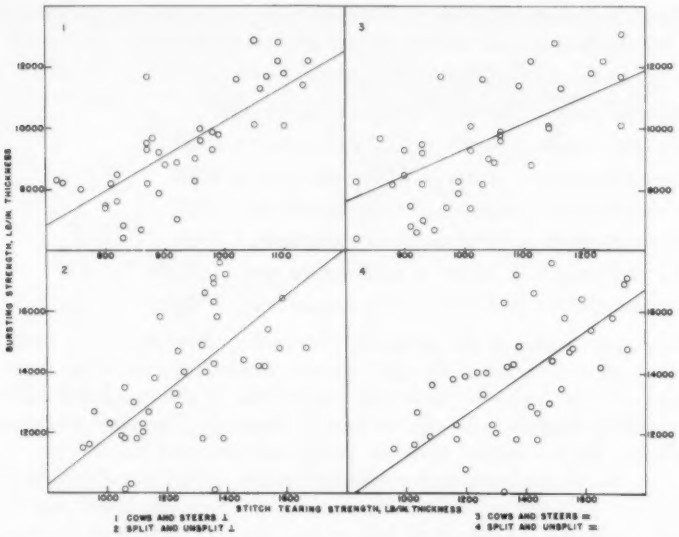

BURSTING STRENGTH, LB/IN. THICKNESS
STITCH TEARING STRENGTH, LB/IN. THICKNESS
1 COWS AND STEERS ⊥
2 SPLIT AND UNSPLIT ⊥
3 COWS AND STEERS =
4 SPLIT AND UNSPLIT =

FIGURE 11. Variation of the stitch tearing strength with the bursting strength for four different groups of leather specimens.

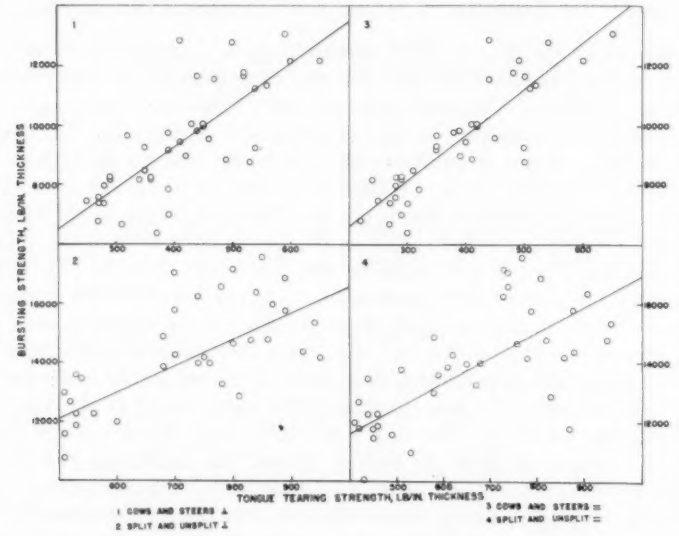

BURSTING STRENGTH, LB/IN. THICKNESS
TONGUE TEARING STRENGTH, LB/IN. THICKNESS
1 COWS AND STEERS ⊥
2 SPLIT AND UNSPLIT ⊥
3 COWS AND STEERS =
4 SPLIT AND UNSPLIT =

FIGURE 12. Variation of the tongue tearing strength with the bursting strength for four different groups of leather specimens.

Some positions show definite weaknesses or definite strengths, which appear to be peculiar to the test with which the strength was determined. For example, burst strength in positions 7 and 17 is low for all sets of results. Tongue tear is generally low in these positions also. The row of blocks next to the backbone, 1 to 7 inclusive, are in general weak in bursting strength. On the other hand, positions in the third row, blocks 21 to 27 inclusive, including the belly center blocks 23, 24, 25, and 26, are high in bursting strength. Block 26 is outstandingly high.

Results for tensile strength, stitch tear, tongue tear, and burst were compared on a unit (equal) hide substance basis. Although results of the density

TABLE 7a

Calculation of Average Tensile Strengths of Split Chrome-Tanned and Chrome-Retanned Cow and Steer Sides to Unit Hide Substance Basis and Results of the Application of Student's "t" Test

	Perpendicular direction				
Location	1 Chrome-tanned*	2 Chrome retanned cow sides†	Difference 2 – 1	3 Chrome retanned steer sides†	Difference 3 – 1
	10 lb/in.²	10 lb/in.²		10 lb/in.²	
1	612	558	— 54	552	— 60
2	645	709	+ 64	627	— 18
3	695	711	+ 16	713	+ 18
4	633	672	+ 39	609	— 24
5	596	673	+ 77	633	+ 37
6	547	604	+ 57	528	— 19
7	718	751	+ 33	698	— 20
11	834	853	+ 19	759	— 75
12	968	1008	+ 40	927	— 41
13	941	944	+ 3	928	— 13
14	799	812	+ 13	763	— 36
15	821	820	— 1	777	— 41
16	693	834	+141	714	+ 21
17	778	797	+ 19	736	— 42
21	635	934	+299	791	+156
22	755	1140	+375	890	+135
23	1099	1047	— 52	1000	— 99
24	968	932	— 36	830	—138
25	968	1031	+ 63	871	— 97
26	829	843	+ 14	772	— 57
27	931	795	—136	696	—235
Average	784	831	+ 47	753	— 31
		t-test 2 vs 1 t = 1.94 (not significant)		t-test 3 vs 1 t = 1.70 (not significant)	

* Results based on averages of 60 specimens.
† Results based on averages of 30 specimens.

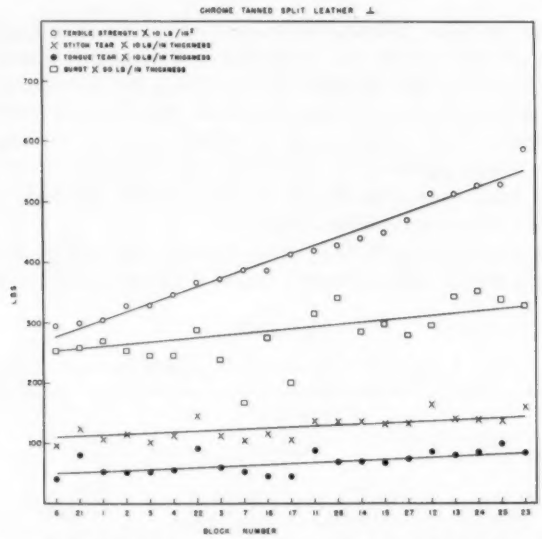

CHROME TANNED SPLIT LEATHER ⊥
TENSILE STRENGTH X 10 LB/IN²
STITCH TEAR X 10 LB/IN THICKNESS
TONGUE TEAR X 10 LB/IN THICKNESS
BURST X 50 LB/IN THICKNESS
LBS
BLOCK NUMBER

FIGURE 13. Comparison of the tensile strength, bursting strength, stitch tearing strength, and tongue tearing strength in the different blocks for chrome-tanned grain split leather tested in the perpendicular direction.

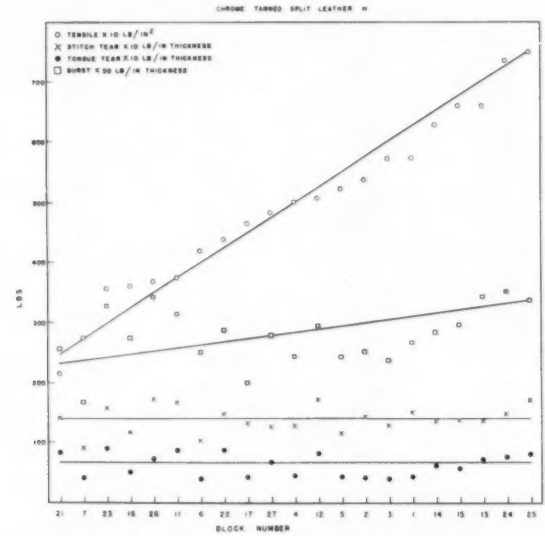

CHROME TANNED SPLIT LEATHER =
TENSILE X 10 LB/IN²
STITCH TEAR X 10 LB/IN THICKNESS
TONGUE TEAR X 10 LB/IN THICKNESS
BURST X 50 LB/IN THICKNESS
LBS
BLOCK NUMBER

FIGURE 14. Comparison of the tensile strength, bursting strength, stitch tearing strength, and tongue tearing strength in the different blocks for chrome-tanned grain split leather tested in the parallel direction.

determinations indicated that there was no significant relation between weight of fiber and strength when different locations were compared, some relation might exist when the same locations of different sides of different tannages were compared. Results for tensile strength, stitch tear, tongue tear, and burst for the split-chrome-tanned leather and for the chrome-retanned cow and steer sides were converted to a unit hide substance basis. Tests for significant differences between the results of the split- chrome-tanned leather and the chrome-retanned cow and steer sides were applied.

These results for tensile strength are given in table 7. They indicate that on a unit hide substance basis there is no significant difference at the 5 per

TABLE 7b

Calculation of Average Tensile Strengths of Split Chrome-Tanned and Chrome-Retanned Cow and Steer Sides to Unit Hide Substance Basis and Results of the Application of Student's "t" Test

	Parallel direction				
Location	1 Chrome-tanned*	2 Chrome retanned cow sides†	Difference 2 - 1	3 Chrome retanned steer sides†	Difference 3 - 1
	10 lb/in.2	10 lb/in.2		10 lb/in.2	
1	1156	978	—178	1078	— 78
2	1062	938	—124	911	—151
3	1072	850	—222	780	—292
4	917	919	+ 2	888	— 29
5	951	1009	+ 58	979	+ 28
6	779	818	+ 39	769	— 10
7	514	599	+ 85	527	+ 13
11	743	777	+ 34	822	+ 79
12	959	1035	+ 76	919	— 40
13	1210	1091	—119	988	—222
14	1139	1018	—121	956	—183
15	1206	1104	—102	1000	—206
16	646	887	+241	757	+111
17	880	848	— 32	760	—120
21	462	803	+341	517	+ 55
22	906	1018	+112	1048	+142
23	668	1211	+543	852	+184
24	1351	1341	— 10	1009	—342
25	1374	1404	+ 30	1110	—264
26	713	1071	+358	795	+ 82
27	957	837	—120	776	—181
Average	936	979	+ 42	916	— 68
		t-test 2 vs 1 $t = 1.01$ (not significant)		t-test 3 vs 1 $t = 2.04$ (not significant)	

* Results based on averages of 30 to 60 specimens.
† Results based on averages of 15 to 30 specimens.

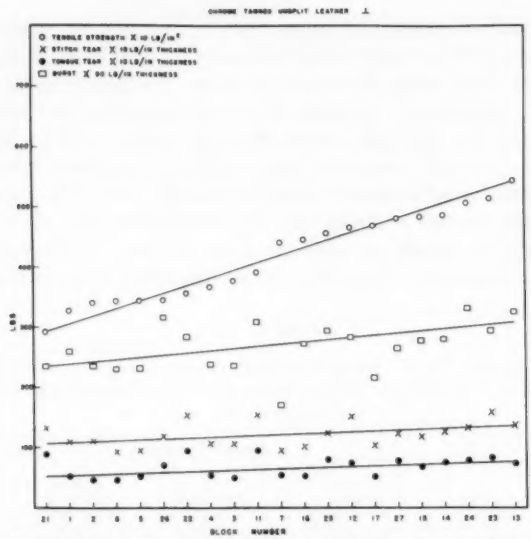


FIGURE 15. Comparison of the tensile strength, bursting strength, stitch tearing strength, and tongue tearing strength in the different blocks for chrome-tanned unsplit leather tested in the perpendicular direction.

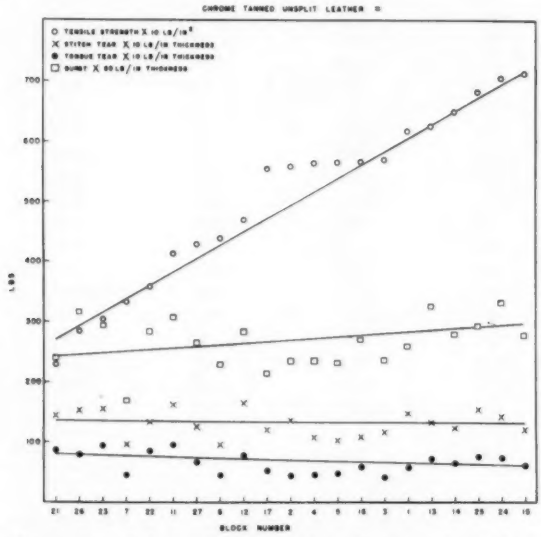


FIGURE 16. Comparison of the tensile strength, bursting strength, stitch tearing strength, and tongue tearing strength in the different blocks for chrome-tanned unsplit leather tested in the parallel direction.

cent level of significance between the tensile strengths of chrome and chrome-retanned leathers. These results agree with those of Roddy [10] who found that the tanning of single fibers did not lower their tensile strength. The results calculated on a hide substance basis show that the strength of the vegetable-retanned leather is just as great as that of the chrome-tanned leather.

The results for stitch tear strength based on a unit hide substance basis for split chrome-tanned and chrome-retanned cow and steer sides are compared in table 8. Three of the four "t" tests applied indicate that a significant difference exists between the stitch tear strengths of chrome-tanned leather and chrome-retanned leather. The stitch tear strengths of the retanned

TABLE 8a

Calculation of Stitch Tear Strength of Split Chrome-Tanned and Chrome-Retanned Cow and Steer Sides on Unit Hide Substance Basis and Results of Application of Student's "t" Test

	Perpendicular direction				
Location	1 Chrome-tanned*	2 Chrome retanned cow sides†	Difference 2 – 1	3 Chrome retanned steer sides†	Difference 3 – 1
	10 lb/in.²	10 lb/in.²		10 lb/in.²	
1	213	177	— 36	210	— 3
2	224	219	— 5	228	+ 4
3	209	210	+ 1	235	+ 26
4	204	215	+ 11	188	— 16
5	183	192	+ 9	192	+ 9
6	178	177	— 1	162	— 16
7	193	215	+ 22	194	+ 1
11	272	263	— 9	250	— 22
12	309	271	— 38	285	— 24
13	256	249	— 7	246	— 10
14	246	228	— 18	224	— 22
15	241	232	— 9	236	— 5
16	208	211	+ 3	198	— 10
17	200	192	— 8	192	— 8
21	264	256	— 8	249	— 15
22	301	276	— 25	282	— 19
23	297	252	— 45	280	— 17
24	253	253	0	259	+ 6
25	259	267	+ 18	269	+ 20
26	263	257	— 6	223	— 40
27	263	218	— 45	215	— 48
Average	239	230	— 9	229	— 10
		t-test 2 vs 1 t = 2.2 (significant)		t-test 3 vs 1 t = 2.62 (significant)	

* Results based on averages of 30 specimens.
† Results based on averages of 15 specimens.

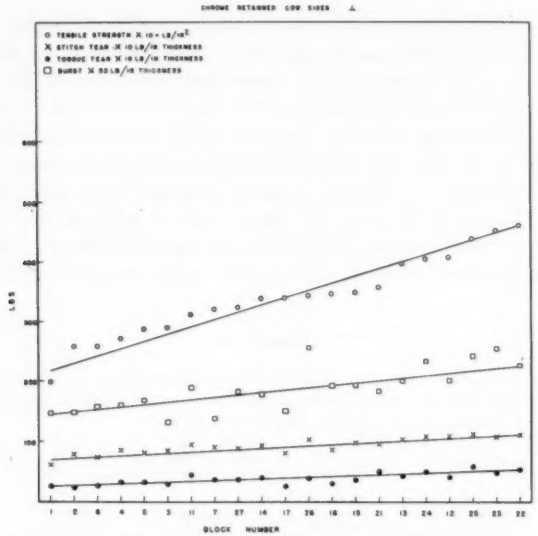

CHROME RETANNED COW SIDES
O TENSILE STRENGTH X 10= LB/in²
X STITCH TEAR X 10 LB/in THICKNESS
● TONGUE TEAR X 10 LB/in THICKNESS
□ BURST X 50 LB/in THICKNESS
LBS
BLOCK NUMBER

FIGURE 17. Comparison of the tensile strength, bursting strength, stitch tearing strength, and tongue tearing strength in the different blocks for chrome-retanned leather from cow sides tested in the perpendicular direction.

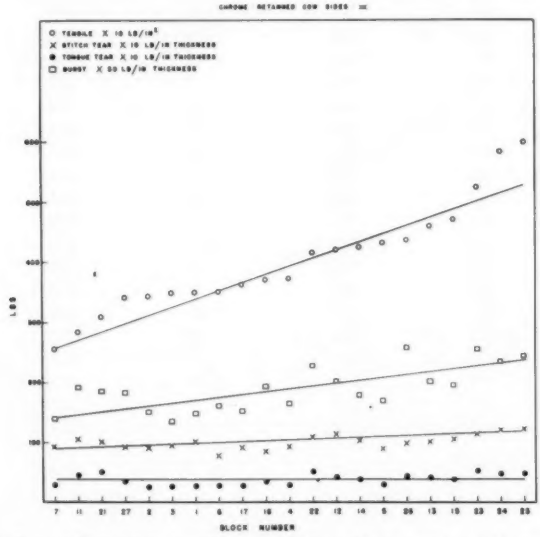

CHROME RETANNED COW SIDES
O TENSILE X 10 LB/in²
X STITCH TEAR X 10 LB/in THICKNESS
● TONGUE TEAR X 10 LB/in THICKNESS
□ BURST X 50 LB/in THICKNESS
LBS
BLOCK NUMBER

FIGURE 18. Comparison of the tensile strength, bursting strength, stitch tearing strength, and tongue tearing strength in the different blocks for chrome-retanned leather from cow sides tested in the parallel direction.

leathers are significantly lower than those of the chrome-tanned leathers. Since there was no significant difference in the results for tensile strength between the two types of leather, the fact that a significant difference is obtained for the stitch tearing strength gives evidence of a difference in the mechanism of the two tests.

The results calculated on this same basis for tongue tear are given in table 9. In this case a highly significant difference (significant at 1-per cent level) exists between the tongue tearing strength of the split chrome-tanned leathers and that of the chrome-retanned leathers. These results are for both per-

TABLE 8b

Calculation of Stitch Tear Strength of Split Chrome-Tanned and Chrome-Retanned Cow and Steer Sides on Unit Hide Substance Basis and Results of Application of Student's "t" Test

	Parallel direction				
Location	1 Chrome-tanned*	2 Chrome retanned cow sides†	Difference 2 - 1	3 Chrome retanned steer sides†	Difference 3 - 1
	10 lb/in.²	10 lb/in.²		10 lb/in.²	
1	306	283	— 23	296	— 10
2	283	249	— 34	250	— 33
3	243	232	— 11	244	+ 1
4	235	230	— 5	236	+ 1
5	212	210	— 2	209	— 3
6	193	182	— 11	185	— 8
7	171	218	+ 47	191	+ 20
11	336	290	— 46	286	— 50
12	328	281	— 47	326	— 2
13	251	240	— 11	248	— 3
14	246	249	+ 3	266	+ 20
15	254	248	— 6	257	+ 3
16	211	206	— 5	205	— 6
17	251	216	— 35	233	— 18
21	302	263	— 39	307	+ 5
22	307	267	— 40	287	— 20
23	297	266	— 31	307	+ 10
24	273	278	+ 5	305	+ 32
25	316	288	— 28	295	— 21
26	336	243	— 93	246	— 90
27	251	228	— 23	230	— 21
Average	267	246	— 21	258	— 9
		t-test 2 vs 1 t = 3.45 (highly significant)		t-test 3 vs 1 t = 1.61 (not significant)	

* Results based on averages of 30 specimens.
† Results based on averages of 15 specimens.

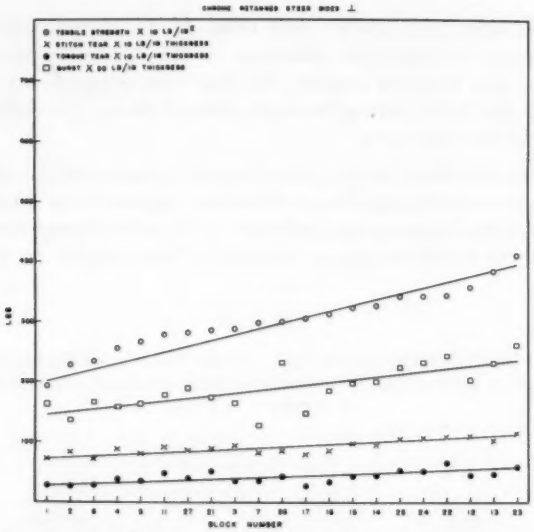

CHROME RETANNED STEER SIDES ⊥
TENSILE STRENGTH X 10 LB/IN²
STITCH TEAR X 10 LB/IN THICKNESS
TONGUE TEAR X 10 LB/IN THICKNESS
BURST X 50 LB/IN THICKNESS
LBS
BLOCK NUMBER

FIGURE 19. Comparison of the tensile strength, bursting strength, stitch tearing strength, and tongue tearing strength in the different blocks for chrome-retanned leather from steer sides tested in the perpendicular direction.

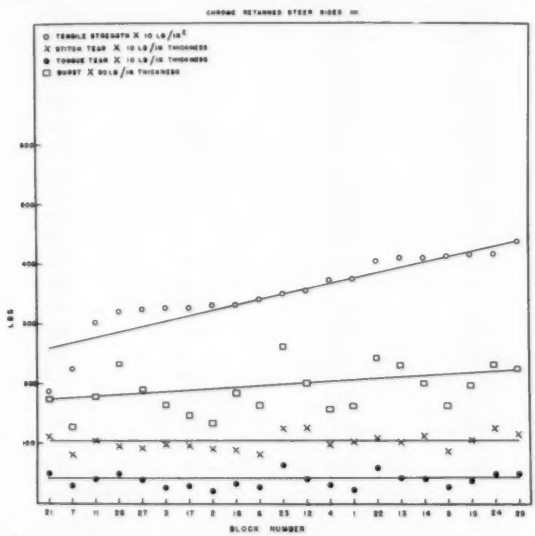

CHROME RETANNED STEER SIDES =
TENSILE STRENGTH X 10 LB/IN²
STITCH TEAR X 10 LB/IN THICKNESS
TONGUE TEAR X 10 LB/IN THICKNESS
BURST X 50 LB/IN THICKNESS
LBS
BLOCK NUMBER

FIGURE 20. Comparison of the tensile strength, bursting strength, stitch tearing strength, and tongue tearing strength in the different blocks for chrome-retanned leather from steer sides tested in the parallel direction.

pendicular and parallel directions of test, and in each test the chrome-retanned leather gives a lower tongue tear value than the chrome-tanned leather.

The same type of results for the burst test are given in table 10. Again, in this test, the differences between the strengths of the split chrome-tanned leather and the chrome-retanned leather are highly significant. The chrome-retanned leather gives lower burst strengths than the chrome-tanned leather.

The results obtained in this comparison of the four different physical tests give further insight into their mechanisms. The principal difference between split chrome-tanned leather and chrome-retanned leather, as shown by the

TABLE 9a

Calculation of Tongue Tear Strength of Split Chrome-Tanned and Chrome-Retanned Cow and Steer Sides on Unit Hide Substance Basis and Results of Application of Student's "t" Test

	Perpendicular direction				
Location	1 Chrome-tanned*	2 Chrome retanned cow sides†	Difference 2 – 1	3 Chrome retanned steer sides†	Difference 3 – 1
	10 lb/in.²	10 lb/in.²		10 lb/in.²	
1	109	76	— 33	83	— 26
2	102	68	— 34	74	— 28
3	112	76	— 36	89	— 23
4	102	84	— 18	93	— 9
5	96	82	— 14	85	— 11
6	76	65	— 11	65	— 11
7	98	91	— 7	86	— 12
11	177	126	— 51	133	— 44
12	162	106	— 56	117	— 45
13	146	107	— 39	113	— 33
14	127	101	— 26	105	— 22
15	124	91	— 33	106	— 18
16	82	77	— 5	80	— 2
17	87	63	— 24	67	— 20
21	173	141	— 32	147	— 26
22	189	137	— 52	168	— 21
23	157	116	— 41	144	— 13
24	156	119	— 37	126	— 30
25	163	141	— 22	137	— 26
26	135	100	— 35	113	— 22
27	146	96	— 50	101	— 45
Average	129	98	— 31	106	— 23
		t-test 2 vs 1 t = 9.76 (highly significant)		t-test 3 vs 1 t = 9.28 (highly significant)	

* Results based on averages of 30 specimens.
† Results based on averages of 15 specimens.

density measurements, seems to be that, in general, the chrome-retanned leather is more plumped. In plumping the leather the fibers are forced apart or separated. It is conceivable that this would lessen the force of attraction between them. The tongue tear strength therefore would be expected to be lower because this test consists of pulling apart fibers. The burst test is also lowered; a similarity in mechanism between burst and tongue tear can readily be visualized, since fibers are also forced apart in the former test. On the other hand, tensile strength, which is a measure of the longitudinal strength of the fibers, has not been affected. Stitch tear, because of the high loads developed before a tear is started, shows smaller differences between chrome and chrome-retanned leathers.

TABLE 9b

Calculation of Tongue Tear Strength of Split Chrome-Tanned and Chrome-Retanned Cow and Steer Sides on Unit Hide Substance Basis and Results of Application of Student's "t" Test

	Parallel direction				
Location	1 Chrome-tanned*	2 Chrome-retanned cow sides†	Difference 2–1	3 Chrome-retanned steer sides†	Difference 3–1
	10 lb/in.²	10 lb/in.²		10 lb/in.²	
1	89	76	— 13	69	— 20
2	83	68	— 15	61	— 22
3	77	66	— 11	69	— 8
4	84	72	— 12	76	— 8
5	80	72	— 8	69	— 11
6	74	65	— 9	65	— 9
7	78	68	— 10	70	— 8
11	175	123	— 52	112	— 63
12	154	103	— 51	109	— 45
13	134	97	— 37	106	— 28
14	112	93	— 19	98	— 14
15	106	89	— 17	94	— 12
16	91	84	— 7	80	— 11
17	81	66	— 15	72	— 9
21	177	130	— 47	138	— 39
22	181	127	— 54	155	— 26
23	170	125	— 45	158	— 12
24	141	110	— 31	121	— 20
25	148	115	— 33	130	— 18
26	143	108	— 35	128	— 15
27	134	86	— 48	99	— 35
Average	120	93	— 27	99	— 21
		t-test 2 vs 1 t = 7.32 (highly significant)		t-test 3 vs 1 t = 6.64 (highly significant)	

* Results based on averages of 30 specimens.
† Results based on averages of 15 specimens.

The results of the different strength tests in the different positions of a side, therefore, are believed to be peculiar to the fiber structure in these positions. Burst and tongue tear tests are considered to be indicative of the forces required to separate the fibers. For this reason burst and tongue tear are low in the butt and backbone areas where the fibers are predominately oriented perpendicular to the grain surface and a force perpendicular to the hide apparently easily separates them. In the belly locations, the fibers run parallel to the surface and a very strong network of fibers is thus developed. For the burst and tongue tear failures in this region more fibers must be severed at the point of rupture. The force necessary for a break in this region is therefore higher.

TABLE 10

Calculation of Bursting Strength of Split Chrome-Tanned and Chrome-Retanned Cow and Steer Sides on Unit Hide Substance Basis and Results of Application of Student's "t" Test

Location	1 Chrome-tanned*	2 Chrome-retanned cow sides†	Difference 2–1	3 Chrome-retanned steer sides†	Difference 3–1
	100 lb./in. thick	100 lb./in. thick		100 lb./in. thick	
1	272	207	— 65	236	— 36
2	250	205	— 45	187	— 63
3	224	164	— 60	205	— 19
4	224	202	— 22	188	— 36
5	223	199	— 24	194	— 29
6	235	186	— 49	187	— 48
7	156	164	+ 8	149	— 7
11	314	263	— 51	242	— 72
12	279	249	— 30	262	— 17
13	315	240	— 75	280	— 35
14	259	216	— 43	233	— 26
15	272	230	— 42	238	—34
16	247	233	— 14	212	— 35
17	190	178	— 12	178	— 12
21	275	243	— 32	243	— 32
22	296	279	— 17	316	+ 20
23	307	296	— 11	319	+ 12
24	323	271	— 52	283	— 40
25	309	286	— 23	287	— 22
26	330	316	— 14	300	— 30
27	277	226	— 51	235	— 42
Average	266	231	— 34	237	— 29
		t-test 2 vs 1 t = 7.5 (highly significant)		t-test 3 vs 1 t = 6.2 (highly significant)	

* Results based on averages of 14 to 16 specimens.
† Results based on averages of 7 to 8 specimens.

Burst and tongue tear ruptures occur at the point and in the direction of greatest weakness. The only test that is well defined directionally is the tensile test. For this reason burst and tongue tears, which result from ruptures in the weakest direction, correlate fairly well with tensile in the perpendicular direction, which is the weakest direction, but very poorly in the parallel direction, which is the strongest direction.

5. Conclusions

As a result of this study it is concluded that the tongue tear and burst tests are highly important in the evaluation of chrome-retanned upper leather. These tests are believed to give an indication as to the forces of attraction between fibers. The tensile test gives an indication of the longitudinal strength of the fibers and for such information appears to be an extremely valuable test. It is believed that the stitch tear test gives an indication of the load supported by the sample before a tear occurs. For this reason it is important in evaluating stitches. After a tear develops, it is similar to the tongue tearing test. Some consideration should be given to the conversion of physical strength data to a unit hide substance basis if the results are to be used for comparing different tannages.

5. Acknowledgment

The author acknowledges the very valuable assistance of John Mandel who suggested methods for the statistical analysis of the data.

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The Chemistry of Vegetable Tannins
XIII Acetyl Values

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In Article VII [1] of this series we have discussed in some detail the validity of the acetyl values of acetylated tannins prepared by us. These values had been questioned by Roux [2] and by Kirby, Knowles and White [3]. Comparative values obtained by acid hydrolysis, alkaline hydrolysis and by trans hydrolysis were presented to show that those obtained by alkaline hydrolysis as employed by us gave reasonable figures.

It was shown that the results given by trans hydrolysis indicated that unacetylated hydroxyl groups were still present whereas the infrared spectrograms indicated the absence of any unacetylated hydroxyl groups.

A further indication of the failure of trans hydrolysis to remove the acetyl groups completely was obtained by deacetylating the acetylated tannins with potassium acetate. Measurement of the acetyl content of the deacetylated tannins by trans hydrolysis gave much lower values than by alkaline hydrolysis. Inspection of the residues from the trans hydrolysis by infrared analysis showed that keto absorption was still present, thus providing a further proof that trans hydrolysis failed to determine the whole acetyl content.

The question at issue has two parts. First, what effect, if any, did the original acetyl impurity in the tannin have on the acetyl value found for the acetylated derivative? Second, is the alkaline hydrolysis method valid and can this be shown by an independent procedure? We can now provide unequivocal answers to these two questions.

Various attempts were made to remove the acetyl impurity from the previously purified quebracho extract A [4]. Long continued liquid-liquid extraction of the aqueous solution with ether gave disappointing results. It was found, however, that the acetyl could be removed by the following method. Fifty grams of quebracho A [1] was dissolved in a minimal amount of acetone and the solution was added dropwise to two liters of chloroform. The precipitate was collected and the process repeated. The product was

*Present address: United States Testing Company, Hoboken, New Jersey.

dried in a desiccator in vacuo. The yield was forty grams. The substance (A265) analyzed as follows:

Found	C	64.27	H	4.67	Ac	None	N	Trace
		64.40		4.92		None		Trace
	$C\text{-}CH_3$	1.92	Ash	0.43	MeO	0.45	Cl	0.44
		1.83		0.28		0.40		0.39

Corr. for ash and av. C 64.57 H 4.81

Calc. $C_{31}H_{28}O_{11}$

C 64.58 H 4.90 $C\text{-}CH_3$ 2.61

It was found to have a maximum absorption in the ultraviolet at 283 mμ showing a density equal to 0.194 in 0.001 per cent solution in absolute ethanol. This gave log E = 4.05 (M.W. = 576.53).

The purified quebracho tannin (A265) was acetylated in the usual manner[4], first by refluxing 2.5 hours with the acetic anhydride mixture followed by treatment of the product with acetyl chloride and dimethylaniline for 40 hours. Purification from methanol and retreatment by refluxing for 3.5 hours with the acetic anhydride mixture followed by chilling from methanol completed the preparation. This acetylated derivative gave the following analysis:

Found	C	61.53	H	4.64	Ac	45.65	Ash	None
		61.72		4.53				None

Calc. $C_{31}H_{17}(O\,Ac)_{11}$

C 61.27 H 4.85 Ac 45.57

It is thus evident that a sample of quebracho, originally containing no acetyl can be converted to a derivative in which all the oxygen atoms have been acetylated. Therefore the original acetyl impurity in the quebracho had no effect on the derivative and was evidently removed during the purification of the acetyl derivative of earlier samples.

In a similar manner, a new purification of wattle was carried out by partitioning it in a mixture of ethyl acetate, acetone and dropping the solution into chloroform. The latter step was repeated. The product showed no acetyl content,* viz:

Found	C	61.61	H	4.75	Ash	None	Ac	None
		61.34		4.75		None		None
$C\text{-}CH_3$	None	MeO	0.44	Cl	0.18			
	None		0.56		0.30			

Calc. $C_{30}H_{26}O_{12} - 0.5\,H_2O$

C 61.33 H 4.63

*The carbon-hydrogen determinations were made by Clark Microanalytical Laboratory, Urbana, Illinois.

The wattle was acetylated thrice in the same manner as quebracho and gave a chalk-white product sintering at 188°C.

Found	C 60.83	H 4.64	Ash None	Ac 47.02
	60.62	4.76	None	46.79
Calc.	C_{30} H_{14} $(O\,Ac)_{12}$			
	C 59.89	H 4.65	Ac 47.69	

The compositions of the acetylated products show that all the oxygen atoms in the tannins have been acetylated. This signifies that all the oxygen atoms exist either in hydroxyl groups or in positions which may become hydroxyl groups under the experimental conditions. Since there may be two rings present containing ether oxygen, we have previously [1] given evidence indicating the possible opening of such rings to form hydroxyl groups. Asahina, Shinoda and Inubuse [5] have clearly shown that the flavanone ring may open during acetylation and the resulting chalcone become acetylated. Coincidentally, Asahina and Inubuse [6] and Narasimhachari and Seshadri [7] have demonstrated that the flavanone ring may open during methylation to form the methylated chalcone.

The evidence presented speaks strongly against the possibility of the tannins being polymers since all the oxygen atoms have been substituted. There still remains some chance of depolymerization of a polymer during acetylation by opening of ether linkages.

The second question concerns the validity of the acetyl determination method. We have already referred to some of the evidence previously presented on this score. It was shown that the alkaline hydrolysis method gave results in consonance with the infrared spectrograms as to lack of unacetylated hydroxyl. It was also shown that the trans hydrolysis method failed to remove all of the acetyl. We now present evidence obtained by the use of radioactive acetyl chloride.

One-half gram of quebracho (A265) containing no acetyl was dissolved in 30 ml. quinoline and 2 ml. acetyl chloride (labeled with C^{14} in the keto group) was added. The mixture was lightly warmed on an infrared lamp to bring all the solid formed into solution and then allowed to stand 66 hours at room temperature. The mixture was poured into excess dilute sulfuric acid, the solid collected and thoroughly washed with water. It was then dissolved in methanol, filtered and chilled from solution. Analysis for acetyl by alkaline hydrolysis gave the following values:

Ac 25.50
25.65

Combustion of the acetylated quebracho and determination of its radioactivity gave:*

> 5.06×10^{-5} millicuries per milligram acetylated quebracho.
> Conversion of the original acetyl chloride to acetanilide and combustion gave:
> 0.0084 millicuries per millimole acetyl chloride. (Average of five determinations with an error of ± 3%)

$$\frac{43 \times 5.06 \times 10^{-5} \times 100}{0.0084} = 25.9\% \text{ Ac}$$

The acetyl value computed by measurement of the radioactivity is in excellent agreement with the value obtained by alkaline hydrolysis. The claim that alkaline hydrolysis degrades the tannins and thus gives rise to a high acetyl value has been shown to be untenable for two reasons. First, both quebracho and wattle have been completely freed of acetyl impurity. It would not be possible to obtain a null value if the alkaline hydrolysis for acetyl analysis produced acetic acid. Second, independent values obtained by alkaline hydrolysis and the measurement of radioactivity gave concordant figures. This would likewise be impossible if alkaline hydrolysis degraded the acetyl derivatives to give high acetyl values.

Summary

1. Quebracho and wattle have been freed of acetyl impurity and then acetylated to their maximum theoretical values.

2. Quebracho has been acetylated with radioactive acetyl chloride. The acetyl value obtained by measurement of the activity of the product was found to check with the value obtained by alkaline hydrolysis.

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*The measurements of radioactivity were made by Tracerlab, Inc., Boston, Mass.

ABSTRACTS

A Rapid Sole Leather Tannage. By G. E. Benskin and A. Chesire. *J. Soc. Leather Trades' Chemists,* **38**, 329 (1954). A brief review of the history of attempts to develop rapid tannage of sole leather is given. A rapid pit tannage for sole leather is described in which the butt can be tanned in ten to fourteen days and the offal given a conventional tannage with the run off liquor from the butt pits. Iso-electric pelt is brought into warm strong tanning liquors of 150° to 190° Bk. at 30-40° C. The tannage takes place without shrinkage or hydrolysis, a heavy hide needing about one week to become struck through and two weeks to attain maximum weight. Mechanical means are required to sink the pelt in strong extract during the first day. All pelt was delimed to the iso-electric point in solutions containing three per cent ammonium chloride on the pelt weight and kept at pH 4.7-4.9 by frequent additions of hydrochloric acid. Study of the effect of acid was made by immersing pelt in solutions of 0.1 , 0.3 , 0.5 and 0.7 *N* citric acid and keeping pelt in these acid baths for two days at 37° C. before tanning. All the pelt which was acid treated before tanning was seriously damaged, showing disintegration. It was conclusively shown that, for success or rapid tannage in hot, strong liquors, it is essential that the pelt be iso-electric on entering the warm liquor. Further study showed that the presence of salt, in the acid treatment before tannage, does not prevent the damage from acid in hot liquors. Increased acidity in the tannage liquor itself did not greatly affect the tannage; but the pH is fairly critical in that tan fixation falls off rapidly above pH 3. To maintain this pH it is necessary to start with lower pH, say about pH 2.85. Temperature plays an important part. Above 40° C. there is danger of damage, which is very definite at 50°. For the blend used, at about 185° Bk., 37° C. proved to be ideal. It is desirable to employ the highest temperature consonant with the production of sound leather. It appears that salts play a much smaller part in determining the physical properties of leather than in usual tannage. The proportion of pyrogallol to catechol tannins is of paramount importance in that the pyrogallol tannin must not constitute too high a proportion. Mixtures of myrobalans and mimosa showed that 30 per cent of the former was safe, 40 per cent doubtful and 50 per cent or more definitely harmful. Use of the run-off liquor for offal tannage is described. Data covering the chemical and physical characteristics of the leathers made are given. Dry hides tend to give leather which is flannelly and having greasy flesh. This process is based on osmotic pressure difference, which causes a rapid diffusion of the water from the pelt, thereby creating a vacuum into which the tanning extract is forced. For such a process completely hydrated pelt is necessary. It is difficult to completely hydrate dry hides and consequently they do not tan so well by this process. R.H.T.

Mixed Indicator for Soda Ash Titrations. by M. Greene. *Chemist Analyst,* **43,** 91 (1954). Because of the difficulty in reaching an exact end-point when using methyl orange as indicator in the titration of a carbonate solution, the author recommends the use of a mixed indicator. This consists of 6 volumes of 0.1 per cent solution of thymol blue, one volume of 0.1 per cent solution of cresol red, and 14 volumes of 0.1 per cent solution of bromcresol green. Six to eight drops of indicator are used for each 100 cc. of carbonate solution. Color change is from pea green to yellow at the end point.

A Color Test for Borates. By C. M. P. Wirth. *Chemist Analyst,* **43,** 101 (1954). Add 5 drops of H_2SO_4 or HCl to the sample solution, mix well, and then add about 1 ml. of a dilute aqueous solution of polyvinyl alcohol. Mix well, and add 1 to 2 drops of a 0.1 *N* iodine solution. If borates are present in moderate concentration (about 6 mg. of H_3BO_3 in 5 ml.), the solution will become blue in color. In low concentrations of borates, the

solution will appear red to red violet. If the order of the reagents is reversed, that is, the iodine solution is added first, followed by H_2SO_4 and polyvinyl alcohol, a green color develops. Substances such as chlorobutanol, dextrin, glucose, and zinc sulfate do not interfere with the test.

Etching Calibraton Marks on Glass. By P. A. Duling. *Chemist Analyst,* **43,** 107 (1954). Well defined calibration lines can be etched on glass by covering the area with pressure sensitive adhesive tape, cutting with razor the required lines and then applying a few drops of hydrofluoric acid to each cut. After 30 minutes, the acid is washed off and the tape removed. Thin, sharply defined lines can be obtained by this procedure.

Cattle Grubs. By F. O'Flaherty. *Lea. Mfg.,* **71,** No. 12, 7 (1954). The author first reviews the life cycle of the two classes of cattle grubs, and also shows this diagramatically. This is followed by a review of the various new treatments for grubs. Internal treatments appear to offer much promise. Among the materials are phenothiazine, di-n-butyl tin dilaurate and hexachlorethane which were used in England. The results were very encouraging. The U. S. Dept. of Agriculture also reported good results with phenothiazine and hypodermic injections of lindane. The Department also reported good results with diazinon, chlorthion and dialkylphosphate. The work will be repeated to determine the effects of chemical treatments on milk and meat. It reports further on the intake by cattle of phenothiazine during the months when heel flies deposit eggs on the legs of cattle. The drug is absorbed into the animal's blood stream so it can attack grubs before extensive damage has been done. The treated cattle showed 80 per cent fewer grubs than untreated animals. Although washes and sprays containing rotenone and lindane are successful in killing the grubs while still present in the hide of the host, the damage to the hide has already been done. Such treatment reduces cattle infection usually within a limited area only.

The University of Cincinnati made a preliminary test on a small number of cattle wherein they were fed from January through April with feed containing a small amount of antibiotic. The cattle did not show grub infestation. European and American investigations indicate that the year-round feeding free-choice of a mixture of one part by weight of phenothiazine to nine parts of salt plus mineral, helps materially in reducing the number of grubs. The U. S. Dept. of Agriculture reports on tests similar to the above where a reduction was effected of 82 per cent in grubs that encysted and cut holes in the thickest part of the animal's hide. Other tests are in progress but they need further testing to determine their toxicity and other factors before they can come into use. The phenothiazine method, however, offers ranchers and cattlemen a practical procedure for grub reduction.

The Evaluation of Sole Leather. By W. Petrie. *Lea. Trades Rev.,* **108,** 60 (1953). The shoe repairman, the tanner, and the shoe manufacturer should pool their knowledge of leathers and supplement this by chemical analysis. The collective information should be beneficial to each and result in a much better product.

Synthetic Resins for Leather Finishes. By W. O. Nutt. *Lea. Trades Rev.,* **108,** 62 (1953). An article on acrylic resins in emulsion form as leather finishes.

Porous Plastic Uppers. *Lea. Trades Rev.,* **109,** 58 (1954). The British Boot, Shoe and Allied Industries Research Association examined plastic shoe upper material which had been perforated by passing an electric spark discharge through it. The holes were spaced about 0.125 inch apart and were approximately 0.1 mm. in diameter. At a relative humidity of 65 per cent on one side of a diaphragm of this material and zero relative

humidity on the other side, the perforated plastic transmitted about 0.06 mgms. of water vapor per square cm. per hour. By comparison, normal upper leather transmitted 3 to 10 mgms. /sq. cm./hr. Since sweat is produced by the upper surface of the foot at from 2 to 10 mgms. /sq. cm./hr. it is evident that, at best, the plastic would transmit only about 5 per cent of the moisture produced by the normal foot. To make the plastic more porous would certainly decrease its strength and affect its appearance. The sample examined was far less waterproof than normal upper leather.

Thoughts About Suspender Liquors. By W. Petrie. *Lea. Trades Rev.*, **113,** 293 (1954). A general discussion on rocker tannage of sole leather.

New Products Developed Since November, 1953. *Amer. Dyestuffs Report,* **43,** 805 (1954). There are listed about 380 dyes, 360 chemicals and 20 pieces of equipment which have been placed on the market during the past year. Among the chemicals are included waxes, sequestering agents, dyeing assistants, bleaching agents, detergents and emulsifying agents. An abstract, originating with the manufacturer, describes each product.

Water Solubles in Insole Leathers. *Lea. Trades Rev.*, **113,** 66 (1954). The present British specification for insole leather requires that the water solubles will not exceed 25 per cent and that the soluble salts measured as sulfated ashed water solubles, shall not exceed 2 per cent. It is now proposed that the latter figure should be raised to 3 per cent. After analyzing over 700 samples of insole leathers and 400 samples of insole leathers from worn shoes, the results showed that 66 per cent of the leather samples and 91 per cent of the wear-complaint shoes had a water soluble ash content of 3 per cent or above, calculated as sulfate salts. The analyses further showed the high percentage of unsatisfactory leathers and secondly that the 3 per cent water solubles ash calculated to sulfates is generally tolerable. The new specification is to operate for an experimental period. The British Boot, Shoe and Allied Trades Association, in addition to the above recommendation, proposes to embark on a research program on leather dyeing and finishing. This program is necessary since different types and combinations of finishes require different treatments in the show room, and the selection of suitable cleaners and dressings for each leather is no easy matter for the foreman. Lakes are particularly difficult to fix or to restore.

Tannery Progress from Fifty Years Ago. By W. Petrie. *Lea. Trades Rev.*, **108,** 331 (1953). The introduction of machinery and mechanical appliances has materially reduced the man power requirements in tanneries. Formerly 150 men processed 750 hides per week, but today the same number of men will process 3000. Further reduction is still possible. The author spells out the man power saving by tannery departments.

Properties of Zirconium Tannin. By A. F. Carter and J. W. Harrison. *Lea. Trades Rev.*, **108,** 334 (1953). Basic zirconium sulfate is a better tanning agent than either the chloride or nitrate. The best liquors have a basicity of 50 or less. It is thought that zirconium combines with hide substance through hydrogen bondage between the hydroxyl groups of the hydrolyzed basic zirconium sulfate and the polypeptide chains rather than by salt linkages with the side chains, as is probably the case with chrome tanning. This theory is supported by the fact that zirconium tanned leather can be retanned with chrome up to the theoretical maximum needed for salt link saturation. Such combination tannage gives a fuller, plumper leather. Used alone zirconium tannage requires 12-15 per cent ZrO_2 on the dry hide weight as against 6 per cent of Cr_2O_3. Straight zirconium tanned leather has a shrinkage temperature of 95° C. with 15 per cent ZrO_2 fixation.

In tanning it is customary to bate the stock heavier and to bleach and pickle in formic acid. Stock is drummed in 200 per cent water, 20 per cent of zirconium salt containing 25 per cent ZrO_2, 10 per cent salt and 1 per cent glacial acetic acid. Time of drumming is usually 4-5 hours. Stock is left in the drum overnight, then neutralized with bicarbonate to pH 4.0, after which it is drained and washed. This is followed by a further neutralization with hypo to pH 4.5 - 5.0 and subsequent washing. Combination tannage with chrome is carried out simultaneously with the zirconium, but final neutralization reaches a pH of 5.0 - 5.5. Zirconium leather needs more oil than chrome. The fatliquors usually consist of raw oils blended with nonionic emulsifying agents to which it is then possible to add sulfated oils up to 50 per cent in some cases. Drying must be done carefully, it being customary to hang goods at room temperature before they enter the drying chamber. Here the temperature should start low and gradually increase, while the reverse is true for the humidity.

Leather Design—A Neglected Art. By C. H. Spiers. *Lea. Trades Rev.*, **108,** 707 (1953). A general discussion on leather surface treatment.

City and Guilds of London Institute Examination Questions. *Lea. Trades Rev.*, **109,** 319 (1953). A set of questions given to candidates sitting for examination in tanning and dressing of heavy and light leather.

Leather Industry Encylopedia. By D. Woodroffe. *Lea. Trades Rev.*, **110,** 329 (1953). A continuation of terms in tanning and their explanation.

Preparation of Crocodile Skins. By I. Mann. *Lea. Trades Rev.*, **110,** 557 (1953). Many crocodile skins are lost to the trade by improper curing. In fact such skins require more care in curing than cattle hides, or goat and sheep skins. Since the belly part of the skin is the only marketable part, it must be protected during the killing and subsequent handling. Certain precautions must be taken to insure a good skin. These are as follows: the carcass should not remain in the water for more than 8 to 10 hours. When it is removed from the water it should be dragged with its back down. No time must be lost between flaying and salting, and in no case shall the skin be dried in direct sunlight. Proper flaying is shown digramatically. There are two saltings - the second about two hours after the first when the skin has had a chance to drain, and additional flesh scraped off. Only fine salt should be used in both saltings, and should be well rubbed in. The skins are then removed to a go-down or store house, rolled with scales inward, with abundant salt for transportation. During transportation the skins should be covered and never piled more than three high. Curing is carried out in a cool shed and is the most difficult operation. Red heat is prevented by adding a preservative, such as santobrite, to the salt. The curing shed should be cool, high ceilinged, properly cross ventilated, and preferably with cement floor. About 3 weeks are required for curing.

Staining of Aniline Dyed Leathers. *Lea. Trades Rev.*, **114,** 568 (1954). Shoe uppers, made with aniline dyed leather, are prone to stain if shoe linings are made of cloth containing more than 5 per cent organic solubles (by hot extraction), 3 per cent organic solubles (by warm extraction) and one per cent soluble inorganic salts. To keep aniline dyed leathers clean, it is recommended that a thin, continuous wax film be spread over the surface of the leather. If shoes are dirty or muddy, they should be wiped off gently with a moist sponge and then with a dry cloth. When they are dry, shoe polish or cream should be applied carefully and evenly.

Sulfite Cellulose Extracts. By W. Petrie. *Lea. Trades Rev.*, **114,** 569 (1954). Sulfite cellulose can be used advantageously with catechol tannins in a yard, wheel extract, or dry dip blend. They are particularly useful as loading material as they do not oxidize or discolor the leather. Most of the commercial products are either soda or ammonia neutralized. Those from Sweden are usually neutralized with ammonia. It is regarded as premature to judge the quality of the syntans having a sulfite cellulose base.

United States Requirements of Tanning Materials. *Lea. World,* **46,** 618 (1954). In 1953 the United States imported tons of extracts as follows: mangrove 3320, quebracho and urundy 119,620, chestnut 9470, valonia 900, wattle 16580, others 1340. Crude tanning materials imported in tons were as follows: myrobalams 9740, valonia 6440, and wattle 11,850. No figures are given for mangrove bark imports.

Absorptiometric Colorimetry in the Textile Laboratory. By C. H. Giles and I. S. Shaw. *J. Soc. Dyers and Colourists,* **69,** 481 (1953). This is a description of the principal uses of absorptiometers and colorimeters in a textile laboratory, including their employment for the determination of the concentration of dyes in solution and of dyes and certain auxiliary products on the fiber, the amounts of metals present on the fiber, and the pH values of aqueous liquids. Tabulated descriptive details of current British instruments are included, and recent developments in the understanding of the relative accuracy attainable in absorptiometric analysis, and its improvement are discussed. The paper is largely a review, but a little new investigational work has been included where necessary to supplement the published information.

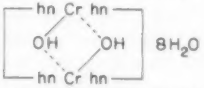
Solubility of Feather Keratin. By. A. M. Woodin. *Nature,* **173,** 823 (1954). Through *J. Soc. Dyers and Colourists,* **70,** 378 (1954). Results of X-ray analysis, dialysis, and chemical reactions of solutions and products obtained from the dissolution of feather keratin in urea-bisulfite solutions are presented, and indicate that the keratin molecule has a molecular weight of 10,000.

The Solubility of Wool in Urea-Bisulfite Solution and Its Use as a Measure of Fibre Modification. By K. Lees and F. F. Elsworth. *J. Soc. Dyers and Colourists,* **70,** 354 (1954). The authors had in mind the possibility that the effect of alkaline treatment on the solubility of wool would be more clearly revealed by the use of a more powerful protein dispersing agent than 0.1 *N* sodium hydroxide as used in the alkali solubility test. It had already been shown by Jones and Mecham [Biochem. 3,193 (1943)] that the keratin-proteins can be partly dispersed in a neutral solution of protein denaturant, e.g. urea, in the presence of a reducing agent, e.g. sodium bisulfite, capable of splitting the disulfide linkages, an essential preliminary reaction in the dispersion of the keratins. The effects of different treatments on the amount of wool dissolved in one hour by a solution containing 50 per cent of urea and 3 per cent sodium bisulfite at 65° C. are shown in tabular form. The method shows promise as a means of indicating the nature of the treatment accorded a sample of wool and is also a sensitive method for assessing the severity of such treatment.

A Study of Certain Natural Dyes. I. Adsorption of Brazilwood and Logwood Coloring Matters by Fibers. By F. M. Arshid, J. N. Desai, D. J. Duff, C. H. Giles, S. K. Jain and I. A. Macneal. *J. Soc. Dyers and Colourists,* **70,** 392 (1954). The adsorption properties of haematein, the coloring matter from logwood, and its leuco compound, haematoxylin, together with brazilein, the coloring matter from brazilwood, have been studied on several types of fibre. Brazilein and haematein are weak acids and are adsorbed by

wool or nylon appreciably only from acid solutions, probably by hydrogen bonding between the fibre and the undissociated haematein or brazilein molecules, as well as by acid-base combination with the charged amino groups in the fibre. Very high adsorption values observed at about pH 1.0 may be indicative of some hydrolytic breakdown of the fibre. In solutions less acid than about pH 7, adsorption is very low on account of the mutual repulsion between the coloring matter anions and the negatively charged fibre. Haematoxylin is a tetrahydric phenol and has the very unusual property, in a single substance, of appreciable substantivity for cellulose as well as for cellulose acetate, nylon, and protein fibres. A study of the apparent heat of adsorption, of the nature of the isotherms, and of the affinities leads to the conclusion that its adsorption by cellulose acetate, nylon, and wool is most probably attributable to the formation of hydrogen bonds with the fibre, those formed by nylon and wool being stronger than those formed by cellulose acetate. The adsorption by cellulose, studied by the same means, appears to be of a different nature and to be attributable to weaker forces, probably van der Waals attraction. Since haematoxylin, with four strong hydrogen-bonding groups, does not seem to form hydrogen bonds with cellulose in water, it is unlikely that any dyes can do so, and their adsorption may therefore be due to physical attraction and not to hydrogen bonding. Some evidence is given that haematoxylin may act as a cross-linking agent between the peptide groups in the molecular chains of proteins. Analytical methods for the coloring matters are evaluated. Refractometry was used in determining the adsorption of haematoxylin. A simple and effective method of packing loose fibres for adsorption tests is described.

A Study of Certain Natural Dyes. II. The Structure of the Metallic Lakes of Brazilwood and Logwood Coloring Matters. By F. M. Arshid, R. F. Connelly, J. N. Desai, R. G. Fulton, C. H. Gilles and J. C. Kefalas. *J. Soc. Dyers and Colourists*, **70**, 402 (1954). The lake-forming reactions of the oxidized and reduced forms of the logwood and brazilwood coloring matters with the more important mordanting metals have been investigated. The oxidized forms appear to be the lake-forming species, the o-hydroxyquinone system of the molecule chelating with the mordanting salt before lake formation can take place. When a chromate or a dichromate is used as mordant, this is first reduced to the chromic cation, at the expense of the total destruction of some of the coloring matter, before lake formation occurs. The logwood coloring matter, when treated with a chromium salt in solution or on the fibre, forms a deep blue 2:1 (dye:metal) complex, which is preceded by a transient purple 1:1 complex. Aluminium, cobalt, and (ferric) iron also form 2:1 complexes, and copper appears to form a 1:1 complex. The lakes formed in substance are amorphous, infusible, and almost insoluble in all solvents. Exact characterisation of these bodies, which appear to be mixtures, is not practicable, but by a consideration of probable reaction sequences, in conjunction with the elementary analyses, a series of formulae have been drawn up which are believed to represent their most probable constitutions. The development of insolubility is believed to be due to one or more of the following effects - mutual precipitation of catonic or anionic lakes by complex anions or cations respectively, formation of non-ionic lakes, and the form of condensation polymerisation known as "olation". It is suggested that similar changes occur in the fibre during the normal mordanting and dyeing processes, and that parallel reaction sequences may take place in dyeing other forms of mordant dye. The deep blue-black logwood lake on the fibre is believed to be a mixture of an anionic complex, e.g. $[hnCr(OH)_2hn]^-$ $[Cr(OH_2)\ (H_2O)_4]^+$, $4H_2O$ with the derived olated body -



(hn=Haematein anion)

The iron lake is probably similar in constitution. In addition to the action of the chelating groups, the phenolic groups not in the quinonoid nucleus of the coloring matter molecule appear to take part also in salt formation with cobalt or copper, but not with chromium or iron.

Adsorption of Water Vapor by Proteins. By H. B. Dunford and J. L. Morrison. *Canadian J. Chem.*, **32,** 558 (1954). Through *J. Soc. Dyers and Colourists*, **70,** 427 (1954). Bull's isotherms for the adsorption of water vapor by proteins are fitted by the Brunauer-Emmett-Teller equation for the range of relative vapor pressures 0.05-0.5. The Harkins-Jura equation is applied to Bull's data, and in most cases linear plots are obtained over the range 0.5 or 0.6 to 0.95. Nylon gives linear plots over the range 0.7 to 0.95. The amount of water vapor in a mono-molecular layer is calculated from both equations and good agreement obtained. It is suggested that a film at least two molecules thick is formed, the upper layer being condensed and occuping the same surface area as the underlying mono-molecular layer, the same number of adsorption sites being involved. The fact that nylon obeys the H.-J. equation over a smaller pressure range may be due to the larger water repellent hydrocarbon spacings between peptide links hindering condensed film formation.

Structure for Collagen. By F. H. C. Crick. *J. Chem. Phys.* **22,** 347 (1954). Through *J. Soc. Dyers and Colourists*, **70,** 476 (1954). A suggested structure consists of two polypeptide chains helically wound around a common axis. The backbones of these chains are related by a diad coinciding with the fibre axis. The asymmetric unit consists of two residues, A and B. All the peptide links have the cis-configuration. The planes of successive peptide links on either chain are alternately perpendicular and approximately parallel to the fibre axis. The two Chains are held together by hydrogen bonds perpendicular to the fibre axis and passing very close to it. The model imposes certain restrictions on the side chains. Residues of type B cannot be proline or hydroxy proline; those of type A can be. There is close approach between the β C atom of a residue of type B on one chain and that of the nearest residue of type A on the other chain, which suggests that one member of any such pair will be glycine.

Thermoelectric Potentials of Dye-Gelatin Phosphors. By L. Gombay. *Kolloid Z.* **133,** 40 (1953). Through *J. Soc. Dyers and Colourists*, **70,** 476 (1954). The thermoelectric potentials of gelatin dyed with Rhodulin Orange N have been measured at different temperatures and humidities. The dry phosphor gave the highest potential and the strongest phosphorescence. From the sign of the potential and the asymmetric conductivity, dyed gelatin phosphors must be classed as semi-conductors.

New Observations on the Chemistry of *N*-Carboxy α-Amino Acid Residues. By D. G. H. Ballard, C. H. Bamford and F. J. Weymouth. *Nature*, **174,** 173 (1954). Through *J. Soc. Dyers and Colourists*, **70,** 476 (1954). The polymerisation of many *N*-carboxy-α-amino acid anhydrides in highly polar organic liquids is catalysed by soluble anhydrous inorganic salts such as LiCl and NaI. The main products are cyclic polypeptides and derivatives of hydantoin-3-acetic acid. Kinetic studies of LiCl-catalysed reactions of glycine *N*-carbonic anhydride show that the initial rate of reaction is not significantly affected by water and does not depend on the method used for purifying the salt. Acids have a strong inhibitory effect, a trace present initially producing an induction period. The hydantoin-3-acetic acid is a comparatively strong acid and the reaction is self-inhibiting at quite small conversions. At salt concentration >0.1 *N* the rate shows second-order dependence on monomer concentration and is independent of LiCl concentration. The rate falls off with decreasing concentration of LiCl below ca. 0.1 *N*. The catalytic effect of salts is apparently connected

with the presence of an >NH group in the *N*-carbonic anhydride molecule and is attributed to complex formation between the anhydride, or hydantoin, and cation, resulting in the displacement of a proton and the formation of a molecule in which the N atom has basic properties sufficient for reaction with *N*-carboxy-α-amino acid anhydrides. *N*-Carboxy-α-amino acid anhydrides which are not N-substituted react readily in the presence of tertiary bases. The products contain considerable amounts of hydantoin-3-acetic acid derivatives and cyclic peptides. A high polarity of solvent favours hydantoin formation. The results obtained with tertiary bases thus resemble those with Li salts in polar media, and a similar mechanism is postulated.

Copolymerization of *N*-Carboxyanhydrides and Amino Acids. By W. Lautsch, R. Schütze and W. Broser. *Kolloid Z.*, **133,** 1 (1953). Through *J. Soc. Dyers and Colourists*, **70,** 477 (1954). The kinetics of polycondensation of mixtures of the *N*-carboxyanhydride of L-glutamic acid and DL- or D-phenylalanine in different proportions has been followed polarimetrically.

Some Observations on the Histology of Keratin Fibers. By B. Manogue, M. S. Moss and R. L. Elliott. *J. Soc Dyers and Colourists*, **70,** 502 (1954). Sections of several different wool and hair fibres have been given chemical treatments alleged to isolate the "subcuticle membrane". The residues consist of parts of the cuticle, cortex, and medulla. The cortex residue is made up of cortical cell nuclei, fibrils, and thin membranes located at the boundaries of the cortical cells. The membranes are more resistant to treatment with sodium hydroxide solution and sodium sulphide solution than the major part of the cells, but are dissolved by proteolytic enzymes. The membranes are probably the material hitherto termed "intercellular cement".

Sulfonation and Sulfation. by J P. Sisley and P. J. Wood. *Amer. Dyestuff Reporter*, **43,** 741 (1954). The authors deal with the mechanisms of sulfonation, sulfonating agents, the sulfonation of liquids, the action of sulfuric acid on unsaturated fatty acids having one double bond and one unsaturated hydroxy acids, the sulfonation of fatty alcohols, alkanes, alkenes, aromatic hydrocarbons, and finally touch on other methods of fixation of $-SO_3H$ and $-O-SO_3H$ groups. Numerous references are cited.

The Treatment of Tannery Wastes. By M. M. Luckens. *Lea. Manufacturer*, **74,** No. 1, 7. No. 8, 7 (1954). The basic authority for the control of the pollution of water is essentially a state function but since waterways know no political boundaries, the Federal Government and various regional agencies are playing an increasingly important part in the abatement of water pollution. Legislation for the control of pollution varies from state to state. Usually the protection of streams rests with Departments of Health, but other agencies, such as the wild life and fish commissions, likewise have authority over stream pollution. However, all coordinate their efforts, so that the end result is usually uniform.

Criteria used in the abatement of stream pollution are of two types, the first deals with the receiving water, and the second describes the waste discharged. Stream standards are divided into two categories, namely, quality of the receiving water and dilution requirements. Effluent standards are of two types. The first type specifies the degree of treatment of per cent removal of a specific pollutant in the waste to be discharged. The second type defines the strength of the waste to be discharged or the amount of specific substances that can be discharged into the stream. Stream standards take into account dilution and the capacity of the stream to assimilate and handle waste. Effluent standards on the other hand are easy to define and to administer.

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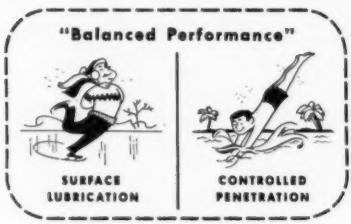

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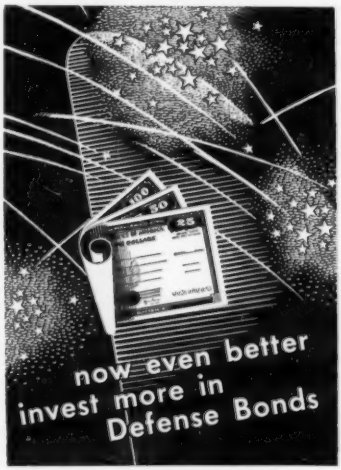

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